

**CASE FILE  
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**THE DEVELOPMENT AND EVALUATION OF  
A COBALT-BASE OXIDATION RESISTANT  
DISPERSION STRENGTHENED ALLOY**

by

**Keki K. Irani**



prepared for

**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION**

**NASA Lewis Research Center**

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**Fred H. Harf and Thomas P. Herbell, Project Managers**

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## FOREWORD

This report was prepared by the personnel of the Buffalo Facility of the Curtiss-Wright Corporation, Buffalo, New York, and describes original work performed under Contract NAS 3-11162.

The contract was awarded to Curtiss-Wright by the NASA - Lewis Research Center. Technical monitoring was provided by the Project Managers, Fred H. Harf and Dr. Thomas P. Herbell, of the Materials and Structures Division of the NASA - Lewis Research Center.

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### ABSTRACT

Co-18Cr-20Ni-4 Vol % ThO<sub>2</sub> powders were prepared by a flash drying selective reduction process starting with an aqueous solution of metal salts and colloidal thoria. Powders were consolidated and extruded into rods with a minimum density of 99% of theoretical. Swaging and annealing studies were conducted to determine the conditions that would lead to a product with high stress-rupture strength. The best process yielded a stress-rupture life of 7.2 hours at 10 KSI (69 MN/m<sup>2</sup>) and 2000°F (1094°C). The alloy recrystallized to a duplex (coarse-fine) structure and thus did not exhibit the desired strength of 3000 hours at 15 KSI (103.5 MN/m<sup>2</sup>) and 2000°F (1094°C).

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## I SUMMARY

The objective of this program was to determine whether a cobalt base dispersion strengthened alloy of acceptable characteristics and properties could be produced from a flash dried solution containing thorium in colloidal suspension. The ultimate objective was to produce a cobalt base dispersion strengthened superalloy with a stress-rupture life of 3000 hours at 2000°F (1094°C) and a 15KSI (103.5 MN/m<sup>2</sup>) load.

In Task I (Processing Studies) the powders were consolidated isostatically, sintered, and then extruded at a ratio of 10:1 at 1094°C. The extruded rods were swaged by four different processing routes. Intermediate annealing was used between the swaging steps. The swaged rods were evaluated in the as-swaged condition and after various heat treatments. The best stress-rupture results in Task I were obtained on a rod swaged at 982°C to 70% R.A. and heat treated for two hours at 1371°C. This procedure yielded a material with a 1094°C stress-rupture life of 7.2 hours at 10KSI (69 MN/m<sup>2</sup>). The microstructural parameters of this material were determined by lineal analysis of a replica electron micrograph. In the as extruded condition the volume fraction, mean free path, and average particle diameter were 4.08 Vol. %, 1.83  $\mu$ m, and 1174Å (10<sup>-10</sup>m) respectively. In the swaged and heat treated condition they were 8.12 Vol. %, 1.72  $\mu$ m, and 2276Å (10<sup>-10</sup>m) respectively.

Task II utilized the best processing route determined in Task I to produce sufficient material for extensive evaluation of the physical and mechanical properties. The results, however, were not up to expectation and the stress-rupture properties were lower than for Task I. The best material from Task II had a 1094°C stress-rupture life of 44.3 hours at 5KSI (34.5 MN/m<sup>2</sup>). The microstructural parameters of this material in the as extruded and as swaged and heat treated conditions were 4.07 and 7.50 Vol. %, 1.60 and 1.48  $\mu$ m, and 1823 and 2016Å (10<sup>-10</sup>m) respectively.

Areal analyses were also conducted on the microstructure of the best materials from Task I and Task II. This analysis was done on replica electron micrographs. For Task I this yielded a median particle diameter of 500 and 250Å (10<sup>-10</sup>m) for the as extruded and as swaged and heat treated material respectively. For the Task II material the results were 1250 and 1000Å (10<sup>-10</sup>m) respectively. An extraction electron micrograph of the as extruded material from Task I was also evaluated by areal analysis. In this case the median particle diameter was determined to be 182Å (10<sup>-10</sup>m).

## II INTRODUCTION

Dispersion strengthening generally improves the high temperature strength properties of metals and alloys, and dispersion strengthened materials have been shown to be useful in applications requiring long time high temperature strength. It has been established that the strongest dispersion strengthened materials have less than five volume percent of stable particles with a particle size of less than 200 to 300 Å ( $10^{-10}$  m) and interparticle spacings in the range of 1-2  $\mu$  m.

Investigation of process development for Co-18Cr-20Ni-4.6ThO<sub>2</sub> alloy by Triffleman and Irani (1) under a previous NASA Contract determined that a Flash Drying Selective Reduction Process using colloidal ThO<sub>2</sub> was suitable for making a dispersion strengthened alloy with acceptable microstructural parameters. Their best procedure resulted in an extruded rod containing a median ThO<sub>2</sub> size of 210 Å ( $10^{-10}$  m) after a 100 hour, 1205°C stability heat treatment. However, Triffleman (2), Preston and Grant (3), and Meijerjng and Druyvesteyn (4), have shown that having fine particles with the correct spacing and volume per cent are not sufficient for obtaining a material with high elevated temperature properties. Furthermore, many investigators have shown that well designed thermomechanical treatments are necessary to obtain a product which is strong and stable at elevated temperatures.

Fraser et al (5) in thermomechanically working Nickel-Thoria used a combined working and annealing treatments to rearrange dislocations into low angle subgrain boundaries rather than annihilate them by full recrystallization treatments. They concluded that the fibered structure appeared to be the most important requirement for maximum high temperature strength of nickel-thoria material and that strain hardening constituted the second main factor contributing to the strength.

Quatinetz and Weeton (6) followed Fraser's work on Ni-ThO<sub>2</sub>. They used 21 cycles of cold rolling and annealing and found that near optimum mechanical properties were obtained at 70% R.A. On the other hand Doble (7) found that he could get superior high temperature strength by recrystallizing after 51% of cold rolling in the longitudinal direction.

Fraser et al (8) investigated the Ni-20Cr-ThO<sub>2</sub> system and obtained good properties at 1094°C and they concluded that a coarse grained microstructure was desirable for attaining high temperature strength.

Mincher and Arnold (9) had demonstrated that for a Co-18Cr-20Ni-2ThO<sub>2</sub> alloy, annealing after extrusion and prior to rotary forging was desirable to produce a high strength material.

The object of this program was to determine whether a cobalt base dispersion-strengthened alloy of acceptable characteristics and properties

could be obtained from a composite alloy - dispersoid powder produced by the Flash Drying Selective Reduction Process. The ultimate goal of this program was to produce a dispersion superalloy with a stress-rupture life of 3000 hours at 1094°C and a 15KSI (103.5 MN/m<sup>2</sup>) load.

To accomplish the objective, the present investigation was divided into two principle areas of study and evaluation; these are listed below:

#### Task I

1. Produce a cobalt base alloy containing 19 - 21 wt. % nickel, 17 - 19 wt. % chromium and 4.4 to 4.8 wt. % thoria (3.8 - 4.2 vol. %).
2. Powders of this composition are to be extruded into rod of substantially 100% of theoretical density. The extruded rod should contain a uniform thoria distribution throughout the matrix with an average size of 0.1 micrometers or less and an average mean free path or interparticle spacing of two micrometers or less.
3. The extruded rod should be given a series of hot working and heat treatment steps to bring the mechanical properties up to the desired goals. Appropriate tests are to be made to determine the effects of these various treatments.

#### Task II

1. Based on Task I a method of processing should be selected to give optimum stress rupture values and the extruded rod should be processed according to this work plan.
2. The processed material should be extensively evaluated for physical and mechanical properties.

### III PROCESS OUTLINE

The process followed in the making of the powder, powder consolidation and extrusion, swaging and heat treatment is shown in step chart form in Figure 1.

The process has been divided into six major sequences.

1. Preparing the oxides (Steps 1 - 4)
2. Reduction Process (Steps 5 - 11)
3. Consolidation (Steps 12 - 15)
4. Extrusion (Steps 16 - 20)
5. Swaging (Steps 21 - 26)
6. Process Selection (Steps 27 - 29)

## TASK I

### IV PROCEDURE FOR PROCESSING ALLOY POWDERS

The method of formulation of Metal Salt - Colloidal  $\text{ThO}_2$  Solutions, Flash Drying, Calcination and Reduction Procedure was chosen on the basis of prior work. (2)

#### A. Preparation of Metal Salt Solutions and Colloidal $\text{ThO}_2$ Suspension

The stock solutions for the various constituents of the alloy Co-20Ni-18Cr-4.6 $\text{ThO}_2$  were prepared as follows:

a) Cobalt Nitrate Stock Solution - The cobalt nitrate stock solution was made by dissolving 1 part of cobalt powder in 7 parts of 45 percent (by weight) nitric acid in a jacketed stainless steel reactor. The solution was filtered and pumped into a stainless steel storage tank. A total of 280 gallons ( $1.06\text{m}^3$ ) of stock solution was prepared; the solution was analyzed at ambient temperature for cobalt (approximately 13.5 wt. %).

b) Nickel Nitrate Stock Solution - In a manner similar to above, nickel powder was converted into 280 gallons ( $1.06\text{m}^3$ ) of a 12.75 wt. % nickel solution.

c) Chromium Nitrate Stock Solution - A weighed amount of chromium nitrate ( $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ) was dissolved in an equal weight of cold water to yield a 6.85 wt. % chromium solution.

d) Thoria suspension - 58g of colloidal  $\text{ThO}_2$  of 50-150 $\text{\AA}$  ( $10^{-10}\text{m}$ ) nominal size were dispersed in one  $\text{dm}^3$  of distilled  $\text{H}_2\text{O}$ .

Weighed amounts of the stock solutions of cobalt, nickel and chromium calculated for form a 35% nitrate salt solution, in which the metals are in proportion of 62Co: 18Cr: 20Ni; were mixed together at ambient temperature in a glass lined jacketed reaction vessel with stirring to form a homogeneous solution.

#### B. Flash Drying

A dual feed system was set up on a Flash Dryer so that a correct proportion of the colloidal  $\text{ThO}_2$  suspension was fed to a line carrying the metal nitrate solutions. The point of juncture of the two solutions was just before the solutions entered the Flash Dryer. The junction was made here to prevent possible precipitation of the colloidal  $\text{ThO}_2$ . The mixed solutions and thoria suspension were flash dried by pumping controlled amount of liquids into a perforated pipe from which droplets fell on a rotating internally heated drum. The drum was surrounded by a stainless steel housing and connected to an acid recovery system. The temperature of the drum was maintained at 200°C. The product was scraped off the drum by a stainless steel "doctor blade".



Sufficient blended solutions were flash dried by the above procedure to yield 6.804 Kg of alloy powder per batch. Typical screen analysis for one of the batches is shown in Table 1. Random batches of flash dried material were checked for analysis by taking 10g samples and reducing in a Leco furnace at 1205°C for 2 hours under cracked ammonia. Table 2 shows the chemical analysis of two typical batches of powder reduced.

### C. Calcination

The flash dried material was then fed into a rotary calciner and heated at 400°C for 15 minutes in air. This step completed the removal of the volatile compounds. The calcined powder was ground in a hammer mill and screened through a 50 mesh screen. Screen analysis of the calcined and calcined and ground material is recorded in Table 3. The ground oxide powder at this stage contained approximately 30 wt. % oxygen and 0.009 wt. % nitrogen.

### D. Reduction Procedures

The oxide powder was selectively reduced by a three step reduction process to alloy plus ThO<sub>2</sub> state. The main objective was a final non-ThO<sub>2</sub> oxygen and carbon of less than 0.03 wt. % each. The details of the procedures are given below.

#### First Reduction

The ground oxides were reduced in a rotary calciner for 30 minutes at 925°C under hydrogen. The partially reduced powder was milled in a hammer mill and screened through 50 mesh screen. The weight loss, oxygen, ThO<sub>2</sub> and screen analysis are shown in Table 4.

#### Second Reduction

At this point sufficient graphite was added to reduce the oxygen to 0.10 wt. % or less. The blending operation was carried out in a 8dm<sup>3</sup> stainless steel twin shell blender.

The blended powders were loaded into perforated boats and reduced in a metal muffle furnace @ 1095°C under hydrogen for 2 hours. The oxygen content on the reduced powders varied from 0.12 to 0.18 wt. % and the carbon from 0.02 to 0.12 wt. %.

#### Third Reduction

The object of this reduction was to further lower the level of oxygen and carbon of the powder.

The boats containing the powder from the second reduction step were not disassembled and the powder was further reduced for 2 hours at 1205°C under dry hydrogen. The sintered powder was crushed in a jaw crusher to approximately minus 4 mesh and then ground in a hammer mill and screened through a 50 mesh screen. The chemical analysis of Co-18Cr-20Ni-4.6ThO<sub>2</sub> Lot No. PP3-17A-69 along with the carbon, oxygen and nitrogen analysis of another Lot No. 13A-76 is recorded in Table 5.

## V CONSOLIDATION

The method of consolidation of powders prior to extrusion was chosen on the basis of prior work. (1) The method consisted in cold compaction of the powder followed by sintering of the green pressed billets.

### A. Cold Compaction

The powders were pressed at 7.5 TSI ( $70 \text{ MN/m}^2$ ) into approximately 2.7" (6.8 cm) diameter X 5.8" (13.92 cm) long billets in an isostatic press. The powders compacted to a density of about 56% of theoretical. The data is recorded in Table 6.

### B. Sintering

A special sintering apparatus was designed for sintering the large billets used in the current investigation. Fig. 2 shows the equipment which consists of a central sintering chamber and a cylinder containing gettering material which is connected to the entrance of the sintering chamber. The gettering material was a coarse powder of 50% Ti - 50% Al. Its main function was to remove traces of moisture and/or nitrogen at sintering temperature from the incoming hydrogen gas before it entered the sintering chamber. The sintering apparatus was loaded into a metal muffle furnace which had cracked ammonia flowing through it. In this manner contamination of the billet during sintering was prevented by having the sintering chamber surrounded by an outside envelope of dry reducing gas. The billets sintered to a density of about 58% of theoretical. The data is recorded in Table 6.

## VI EXTRUSION AND EVALUATION

### A. Extrusion of Billets

The billets from sintering run Nos. 45, 46, 47, 48, 50, 51, 52 and 53 were canned in mild steel cans as shown in Figure 3. The cans were evacuated to one micron ( $0.13 \text{ N/m}^2$ ) absolute pressure and sealed off. The billets were extruded at  $1094^\circ\text{C}$  with an extrusion ratio of 10:1. Other pertinent data on the extrusion variables are recorded in Table 7.

The extruded rods were x-rayed for internal defects and none were found. The extruded rods were decanned in 50% nitric acid. Some of the can material could not be removed by acid and therefore the rods were machined to 0.790" (1.80 cm) in O.D. to remove the stray iron and to have a uniform O.D. for swaging.

### B. Evaluation of the Extrusions

The extruded rod was evaluated for carbon and oxygen, content, hardness, density, microstructural analysis by optical, replica electron-micrograph and electronmicrograph of extracted particles, and room and  $1094^\circ\text{C}$  tensile properties. The data is recorded in Table 8.

#### 1. Density and Hardness

A slice was cut from each extruded rod from the front, center and back and these slices were checked for density and hardness. Density measurements of the extruded rods were made by the water displacement technique. All the rods had densities of 99% of theoretical or more.

#### 2. Chemical Analysis

A chemical analyses for C and O<sub>2</sub> were run on a slice from the center of each extruded rod. Non-ThO<sub>2</sub> oxygen analyses indicated in all cases lower oxygen contents than theoretical. With the technique of oxygen analysis used at present time there appears to be inherent inaccuracies of the ThO<sub>2</sub> and O<sub>2</sub> analysis. Thus it was concluded that even if a perfect reduction was achieved 0.000 wt. % non-ThO<sub>2</sub> O<sub>2</sub> content), the non-ThO<sub>2</sub> O<sub>2</sub> analyses could only be used for comparative purposes. The other method of checking the amount of impurity dispersoids is to examine samples of the extruded rods by electronmicroscopy, at 10,000 X minimum and to carry out a lineal or areal analyses on the resulting micrographs.

#### 3. Structural Analysis

Photomicrographs were made on three slices from extrusion 2 and from the center slice of extrusion 1 of Lot No. 17A-69. Figure 4 illustrates the fine elongated grains (ASTM 10 and finer) typical of the longitudinal cross sections.

Lineal analyses (See Appendix I) were made on representative replica electron micrographs for the front, middle, and back of extrusion No. 2. These electron micrographs are shown in Figure 5, 6, and 7 respectively. The measured microstructural parameters by lineal analysis are included in Table 8. The measured parameters for the middle of the extrusion were: particle volume fraction - 4.08 Vol. %; average particle diameter,  $1174\text{\AA}$  ( $10^{-10}\text{m}$ ); and mean free path,  $1.82\mu\text{m}$ . The median particle diameter by areal analysis (See Appendix I) was  $500\text{\AA}$  ( $10^{-10}\text{m}$ ). An electron micrograph was also made of the particles extracted from the middle section of Extrusion No. 2. This extraction micrograph is shown in Figure 8. The areal analysis of the extracted particles yielded median particle diameter of  $182\text{\AA}$  ( $10^{-10}\text{m}$ ).

Figure 9 show microstructure of extruded rod of lot no. 13A-76 to be used in Task II. At 1000X magnification fine elongated grains (ASTM 10 or finer) can be seen while at 100X magnification scattered white elongated streaks can be observed indicative either of a non uniform structure or that the  $\text{ThO}_2$  particles were too fine to be resolved. Fig. 10 illustrates the electronmicrograph and reveals these white elongated streaks as areas lean in  $\text{ThO}_2$  particles. Lineal and areal analysis was run on the electronmicrograph. Lineal analysis calculations gave a  $\text{ThO}_2$  particles size of  $1833\text{\AA}$  ( $10^{-10}\text{m}$ ) while the areal analysis gave a median particle size of  $1250\text{\AA}$  ( $10^{-10}\text{m}$ ). Thus for the lot no. 13A-76 the  $\text{ThO}_2$  particles in the extruded rod were evidently much coarser than in lot no. 17A-69.

#### 4. Mechanical Properties

Tensile bars were machined from extrusion 2 and tensile tests in air at room temperature and  $1094^\circ\text{C}$  were run in duplicate. The results are included in Table 8. The  $1094^\circ\text{C}$  tensile result was so low that it was deemed useless to run a stress rupture test.

## VII HEAT TREATMENT OF EXTRUSION AND EVALUATION

Two inch (5.08 cm) sections of bars from Extrusion #2 were given 14 heat treatments. Density and hardness were measured on each of the heat treated extruded bars. 1094°C tensile tests were run on some of the heat treated bars. The data is recorded in Table 9. The hardness remained unchanged for heat treatments up to 2 hours at 1371°C and dropped for longer heat treatment times. There is a 50% increase in 1094°C tensile strength for bars heat treated at 1371°C compared to bars heat treated at 1316°C. Therefore stress rupture tests were run on 1371°C heat treated bars. In order that the maximum stress rupture could be determined in a reasonable amount of time, it was decided to increase the stress by 1,000 psi (6.9 MN/m<sup>2</sup>) after approximately 24 hours. The results are recorded in Table 10. The 4 hour heat treatment at 1371°C gave the best stress rupture results. The electronmicrograph from the bar heat treated 4 hours at 1371°C is shown in Figure 11. A lineal analysis of the electronmicrograph gave a particle volume of 6.92 vol. %, an average diameter of 1132Å (10<sup>-10</sup>m) and interparticle spacing of 1.02 μm. This result compared with those in Table 8 of the as extruded bar indicate that there was no growth of ThO<sub>2</sub> particle size thus showing a good stability of ThO<sub>2</sub> on heat treatment at elevated temperature. The electronmicrograph revealed that the ThO<sub>2</sub> particles had spheroidized and also that there was some precipitation of a new phase. This phase was identified by x-ray fluorescence to be rich in chromium.

Because of the detection of new phase in the extruded rod heat treated for 4 hours at 1371°C it was decided to heat treat the extruded rod at a lower temperature of 1316°C prior to swaging.

# VIII SWAGING

A swaging plan was drawn up for the swaging of the extruded bars. The plan is shown in Table 11.

The swaging plan was based on the work of Fraser et al (5) and Quatinetz and Weeton (6). They found that better properties were obtained in the working of Nickel-Thoria when reductions per pass were limited to 5 to 10% of the previous area.

The plan called for swaging 4 bars to 60% R.A. in the 9 steps. Two bars were swaged at 1205°C and 2 bars were swaged at 982°C. One bar at each temperature was given a 2 hour anneal at 1316°C before swaging. Some end face cracking occurred on the 982°C (both in the as extruded and in the as heat treated rods) and therefore these rods were annealed at 1205°C for 20 minutes between swaging passes and then swaged at 982°C. The 1205°C swaged bars (both as extruded and as heat treated) were successfully swaged with virtually no cracking and did not require additional anneals. At 60% R.A. the bars were cut in half and swaged down to 90% R.A. with pieces removed for testing at 70%, 80% and 90% R.A. The swaging schedule was repeated on additional 4 bars (same conditions), the bars were swaged to 60% with samples removed at 20%, 40% and 60% R.A.

The density, hardness and 1094°C tensile results for all the four bars are shown in Tables 12 to 15 and in Figures 12 to 15.

## A. Extruded and Swaged at 982°C

The curve of the tensile results, Figure 12 indicate maximum ultimate tensile strength at 70% and 80% R.A. The bars which had the highest tensile values were selected for stress rupture testing, however the number of tests were limited since only 3 bars were available for 70% and 80% R.A. The 80% R.A. bars gave the best rupture results of 23.5 hours at 7000 p.s.i. (48.3 MN/m<sup>2</sup>). The as swaged microstructure of 60% to 90% R.A. exhibited fine elongated grains of ASTM 9 or finer. The typical structure is shown in Figure 16.

## B. Extruded and Swaged at 1205°C

The curve of the tensile results Figure 13 is not similar to Figure 12 but does seem to show a maximum at 80% R.A. However the stress rupture results are much lower than 982°C swaged material. The microstructure show fine elongated grains of ASTM 9 or finer for 60% to 80% R.A. material and fine equiaxed grains for 90% R.A. material. The typical structure is shown in Figure 17.

## C. Heat Treated at 1316°C for 2 Hours and Swaged at 982°C

This material appears to have maximum tensile properties between 60% and 70% R.A. The high tensile strength at 70% R.A. does not follow the curve. A duplicate test was not run in order to save the bars for further testing.

The stress rupture values for 60%, 80% and 90% R.A. were as low as that of 1205°C swaged material. The microstructures for 70%, 80% and 90% R.A. show a mixture of fine and equiaxed grains. The typical structure is shown in Figure 18.

D. Heat Treated at 1316°C for 2 Hours and Swaged at 1205°C

This material gave a large scatter of results from 70% to 90% R.A. and no definite curve could be drawn in this area. A higher value in tensile was reached at 70% and 90% R.A. but these results could not be duplicated. The stress rupture values were as low as the swaged materials of the previous two series. The microstructures from 60% to 90% R.A. show a mixture of fine and coarse elongated grains. The typical microstructure is shown in Figure 19.

## IX HEAT TREATMENT OF SWAGING AND EVALUATION

The as swaged bars were evaluated for two heat treatments, 2 hours at 1316°C and 2 hours at 1371°C. Initially the heat treated bars were tested for 1094°C tensile to determine if there was a significant improvement and subsequently all the remaining bars were tested in stress-rupture at 1094°C. Only three bars were available for 70% and 80% R.A. in all the four series (A, B, C, D) and a greater percentage of the tests were made on the 60% R.A. and 90% R.A. swaged material where more bars were available.

Density, hardness and microstructural analysis were run on all the heat treated material. The data is recorded in Tables 16 to 30.

### A. Extruded and Swaged at 982°C

The heat treatment at 1316°C did not improve the tensile and stress rupture properties. Examination of the microstructure of 70% R.A. material revealed that the grains had remained very fine and elongated. Heat treatment at 1371°C led to significant improvement in tensile and stress rupture properties. The best stress rupture result was on 60% R.A. material, however this result could not be duplicated. At 1094°C the stress-rupture life was 7.2 hours at 10 KSI (69 MN/m<sup>2</sup>). The 70% R.A. also gave a high stress rupture value of 19.8 hours at 8 KSI (55.2 MN/m<sup>2</sup>). The data is presented in Tables 19 and 20.

The microstructure of the 60% R.A. material as shown in Figure 20, consists of a mixture of coarse elongated grains (ASTM 1 to 4) and fine elongated grains (ASTM 6 to 8). For the 70% R.A. material, Figure 21 the microstructure showed grains of ASTM 5 to 6.

At 90% R.A. the 1094°C tensile strength decreased. Direct comparison of the stress-rupture properties of the swaged and swaged and heat treated material was not possible. The heat treated material had a 1094°C stress-rupture life of 0.1 hour at 8 KSI (55.2 MN/m<sup>2</sup>). The as swaged material was initially loaded at 5 KSI (34.5 MN/m<sup>2</sup>) and ruptured after 6.8 hours at 6 KSI (41.4 MN/m<sup>2</sup>). The structure of the 90% R.A. material after heat treatment was a mixture of fine to coarse equiaxed grains (ASTM 4 to 9) as shown in Figure 22. The equiaxed grains may have resulted from an excessive amount of work per pass in the later stages of swaging. In swaging to about 75% of the original area the reductions per pass were about 10% of the previous area whereas at about 75% of the original area the reductions per pass ranged from 14.5 to 20% per pass due to the lack of swaging dies of proper size. Fraser et al (5) found that better properties were found in the working of nickel-thoria when reductions per pass were limited to 5 to 10% of the previous area.



### B. Extruded and Swaged at 1205°C

Table 22 and 23 show the 1094°C tensile and stress-rupture results respectively and Table 24 gives a descriptive comparison of the microstructure.

The highest 1094°C stress rupture result for this series was obtained on a 80% R.A. piece. This result correlates well with the high 1094°C tensile value obtained. However, the 80% R.A. stress-rupture value was not as high as for the 60% and 70% R.A. pieces swaged at 982°C and heat treated. This may be due to the fact that less cold work was imparted into these samples and also may be correlated with the microstructure Fig. 23 which while showing grain growth to coarse elongated grains (ASTM 1 to 3) also show the presence of large equiaxed grains (ASTM 1 to 3). The formation of the equiaxed grains, as explained earlier, may be due to the higher percentage of deformation during the last stages of the swaging operation.

After heat treatment the grains of the 60% R.A. sample grew to ASTM 5 to 10 Fig. 24 but the structure was apparently not sufficiently coarse i.e. ASTM 1 to 2, to give good stress-rupture properties.

The 70% R.A. sample Figure 25 gave a mixed structure with 60% of structure showing grain growth of large elongated grains (ASTM 1 to 3) and 40% showing fine grains ASTM 8 to 9. The stress rupture was somewhat low. It was decided to heat 70% R.A. pieces for a longer time, 4 hours at 1371°C in order to grow all the grains to ASTM 1 to 3 and hopefully to increase the stress rupture properties. However, the longer heat treatment resulted in a structure consisting of fine elongated grains (ASTM 7 to 8) as shown in Figure 26 and the stress rupture value remained unchanged. We do not have any reason to explain why the grains became finer.

The 90% R.A. heat treated pieces showed a 30% increase in the 1094°C tensile, but the stress-rupture results were lower than as swaged. The lower stress-rupture values may be accounted for by the presence of an equiaxed grain structure as shown in Figure 27.

### C. Extruded, Heat Treated 1316°C for 2 Hours and Swaged at 982°C

Table 25 and 26 show the results for the 1094°C tensile and stress-rupture respectively and Table 27 gives a descriptive comparison of the microstructure.

The ultimate tensile results for the 60% R.A. heat treated piece was 50% to 60% higher than the non heat treated material - i.e. 13.5 KSI (93.15 MN/m<sup>2</sup>) up from 9.8 KSI (67.62 MN/m<sup>2</sup>) or 7.8 KSI (53.82 MN/m<sup>2</sup>) values. The 70% R.A. material had a high ultimate strength i.e. 16.7 KSI (115.23 MN/m<sup>2</sup>), but on heat treatment the strength decreased to 14.9 KSI (102.81 MN/m<sup>2</sup>). However, the stress rupture results of the 60% and 70% R.A. heat treated pieces were lower than the samples swaged at 982°C and heat treated. These lower stress rupture values may be correlated with the microstructure.

In the case of 60% R.A., a large percentage of grains (estimated at 50% had grown to ASTM 5 to 7 and the balance to ASTM 1 to 2. This structure does not meet the criteria set forth by Fraser et al (8), namely the development of a uniform coarse elongated structure.

The lower stress-rupture results for the 70% R.A. material can also be explained by the heterogenous or duplexed nature of structure namely a mixture of coarse to fine, elongated and equiaxed structure.

The 80% and 90% R.A. material had lower stress rupture values. The lower properties can be explained on the basis that the microstructure of these materials showed a predominately equiaxed grain structure.

The microstructures for the 60%, 70%, 80% and 90% R.A. are shown in Figures 28, 29, 30 and 31.

#### D. Extruded, Heat Treated 1316°C for 2 Hours and Swaged at 1205°C

Tables 28 and 29 show the results for 1094°C tensile and stress-rupture respectively and Table 30 gives a descriptive comparison of the microstructure.

The 60% and 90% R.A. material exhibited a slight decrease in 1094°C tensile value compared to specimens swaged at 982°C.

The best stress-rupture results were obtained on the heat treated 80% R.A. material. The higher stress-rupture value correlates well with the development of a relatively coarse elongated structure made up of 50% grain size ASTM 1 to 3 and 50% ASTM 6 to 7.

The 60% and 70% R.A. material had lower stress-rupture values than the 80% R.A. material due to the presence of fine elongated grains of ASTM 8 to 10. The 90% R.A. material is typical of the four series in that it has a fine equiaxed structure and lower stress-rupture values.

The microstructures for the 60%, 70% and 80% R.A. are shown in Figures 32, 33 and 34.

#### E. Lineal and Areal Analysis

Electronmicrographs were made on the samples of the following processes.

- a) Swaged at 982°C to 60% R.A.
- b) Swaged at 982°C to 60% R.A. and heat treat for 2 hours at 1371°C
- c) Swaged at 982°C to 70% R.A.
- d) Swaged at 982°C to 70% R.A. and heat treat for 2 hours at 1371°C
- e) Swaged at 1205°C to 60% R.A.

Lineal and areal analysis were run on the electronmicrographs and the data is presented in Table 31. Electronmicrographs are shown in Figures 35, 36, 37, 38 and 39.

The average thoria volume percent of the eight samples by lineal analysis was 7.63 as compared to the average volume percent of 6.23 by areal analysis. Both these figures are still higher than the theoretical figure of 4.0% volume percent. The average diameter of the particles by lineal analysis of the eight samples is  $1728\text{\AA}$  ( $10^{-10}\text{m}$ ) while the median particle size of the particles by areal analysis is  $343\text{\AA}$  ( $10^{-10}\text{m}$ ).

The results of lineal analysis from Table 31 clearly show a considerable increase in thoria particle size during the thermomechanical working of the extruded bar. The thoria particles have grown from  $1174\text{\AA}$  ( $10^{-10}\text{m}$ ) in the as extruded state to  $2276\text{\AA}$  ( $10^{-10}\text{m}$ ) and  $2102\text{\AA}$  ( $10^{-10}\text{m}$ ) for the swaged at  $982^{\circ}\text{C}$  heat treated 60% and 70% R.A. material. However the results of areal analysis indicate that the thoria particles have remained stable, the median particle size being  $250\text{\AA}$  ( $10^{-10}\text{m}$ ) for both 60% R.A. and extruded material while at 70% R.A. the median particle size had increased to  $500\text{\AA}$  ( $10^{-10}\text{m}$ ).

However the  $1094^{\circ}\text{C}$  stress-rupture properties were relatively high with stress-rupture results of 7.2 hours at 10 KSI ( $69\text{ MN/m}^2$ ) and 19.8 hours at 8 KSI ( $55.2\text{ MN/m}^2$ ) to indicate that the thoria particles were sufficiently fine to contribute significantly to strengthening at elevated temperature.

## TASK II

### X PROCESS SELECTION

Since the 1094°C stress rupture results from Task I were lower than desired, it was decided to swage the extruded rods of lot no. 13A-76 to 70% R.A. by three routes. These were selected to give improved properties.

In Route 1 the swaging procedure used was that which gave best mechanical properties in Task I i.e. swaging at 982°C with anneal at 1205°C. For Route 2, swaging was done at 982°C with annealing at 1094°C and in case of Route 3 annealing and swaging were done at 982°C.

The rods by the three routes were heat treated and evaluated. One process was then selected and the material produced was extensively evaluated for physical and mechanical properties.

#### A. Swaging

All the bars were successfully swaged with only minor cracking on the ends at 40% and 50% R.A. at which point a 1/2" (1.27 cm) piece was cut off and swaging continued. The actual steps used in the swaging and other pertinent data are shown in Tables 32 and 33. Table 34, 35 and 36 show the results obtained in 1094°C tensile, stress rupture and the microstructure of the as swaged rods. The tensile results fall within the range obtained in Task I but the best stress rupture results (Route 3) fall considerably lower than the best results obtained in Task I i.e. 16.7 hours @ 5 KSI (34.5 MN/m<sup>2</sup>) vs. 23.5 hours at 7.0 KSI (48.3 MN/m<sup>2</sup>). Figure 40 shows the microstructure of the material swaged by Route 3.

#### B. Heat Treatment of Swaging

Tables 37, 38, 39 show the results obtained after heat treatment at 1371°C for 2 hours. The tensile results have dropped slightly as compared to the swaged material and are also lower than the best results obtained in Task I. The stress rupture results show a large decrease as compared to the swaged material and also are considerably lower than the best results obtained in Task I i.e. 1 hour at 5 KSI (34.5 MN/m<sup>2</sup>) vs. 7.2 hours at 10 KSI (69 MN/m<sup>2</sup>).

Route 2 and 3 materials were heat treated for 4 hours at 1371°C and tested for 1094°C tensile and the microstructure was examined. The results are shown in Tables 40 and 41. There was no significant increase in tensile strength. Further testing was carried out on Route 1 and 2 material heat treated for one, two and four hours at 1205°C and 1316°C in the hope that lower annealing temperatures would improve the properties. The stress rupture of heat treated pieces of Route 1 were very low. All the samples broke under one hour at 5 KSI (34.5 MN/m<sup>2</sup>).

For Route 2, the maximum stress-rupture value obtained was 6.7 hours at 5 KSI (34.5 MN/m<sup>2</sup>) after a one hour heat treatment at 1316°C.

Route 3 material had the highest stress-rupture strength in the swaged condition, 16.7 hours at 5 KSI ( $34.5 \text{ MN/m}^2$ ). Heat treat experiments were run and the samples tested for stress rupture. Table 41 shows the stress rupture results and the microstructures are described in Table 42. The various microstructures of the heat treated pieces from Route 3 are shown in Figures 41 and 42. Figures 40 and 42 show the microstructures of the specimens swaged to 70% R.A. at  $982^\circ\text{C}$  and swaged to 70% R.A. at  $982^\circ\text{C}$  and heat treated  $10\frac{1}{2}$  hours at  $1205^\circ\text{C}$ . These microstructures are similar to the as extruded structure in that all three have white elongated streaks sandwiched between fine elongated grains. The white streaks were identified in the electronmicrograph, Figure 10 of the extruded rod as areas lean in thorium.

Figure 41 shows photomicrograph of another 10.5 hour heat treated piece of Route 3 which had the highest stress-rupture value of 44.3 hours at 5 KSI ( $34.5 \text{ MN/m}^2$ ). The microstructure reveals a mixture of fine elongated grains and coarse elongated grains of ASTM 6 to 8.

From the large scatter in stress rupture results and inhomogeneity of microstructure it was concluded that the swaged rod did not react in a reproducible manner to heat treatment.

This electronmicrograph lineal and areal analyses were run on the electronmicrograph of material processed by route 3, shown in Figure 43. The material was swaged to 70% R.A. and heat treated at  $1205^\circ\text{C}$  for 10.5 hours. The results compared with that of extruded rod are presented in Table 44. It is evident that the lot no. 13A-76 material in both the as extruded and heat treated state had a much coarser particles with median particle size of  $1250\text{Å}$  ( $10^{-10}\text{m}$ ) and  $1000\text{Å}$  ( $10^{-10}\text{m}$ ) respectively as compared to the swaged and swaged and heat treated material which had particle sizes of  $500\text{Å}$  ( $10^{-10}\text{m}$ ) and  $250\text{Å}$  ( $10^{-10}\text{m}$ ) respectively. Tensile and stress-rupture results in duplicate on the swaged rods, Tables 34 and 35 show consistent properties. It was therefore decided to select rods swaged by Route 3 to test extensively for physical and mechanical properties.

Single tensile tests were made at room temperature,  $649^\circ\text{C}$ ,  $760^\circ\text{C}$ ,  $871^\circ\text{C}$ ,  $982^\circ\text{C}$ ,  $1094^\circ\text{C}$  and  $1191^\circ\text{C}$  and stress-rupture tests in duplicate at three loads at  $982^\circ\text{C}$  and two loads at  $1094^\circ\text{C}$ . The results of the tensile tests are shown in Table 45.

The stress-rupture results are shown in Table 46 and plotted in Figure 44. The plot for stress rupture vs hours at  $982^\circ\text{C}$  assumes the validity of three of the four results obtained and rejects the result of 33.5 hours at 8.5 KSI ( $58.65 \text{ MN/m}^2$ ) and assumes the curve will be linear (on log-log paper) through 10,000 hours. Also plotted on this graph are stress rupture values

for Alloy IN 100 at 982°C and 1094°C for comparison purpose. The 982°C and 1094°C stress levels to give 10,100 and 1000 hours are given in Table 47. The curves on figure 44 indicate that for both 982°C and 1094°C that IN 100 has higher stress rupture value than the Route 3 swaged material.

The x-ray diffraction examination of the Co-18Cr-20Ni-4.6ThO<sub>2</sub> swaged at 982°C by Route 3 showed only the presence of ThO<sub>2</sub> and an F.C.C. structure for the base alloy. The data is recorded in Table 48.

## XI CONCLUSIONS

The evaluation of the extruded and swaged Co-20Ni-18Cr-4.6ThO<sub>2</sub> alloy made by Flash Drying Selective Reduction Process using Colloidal ThO<sub>2</sub> gave the following major results.

### Task I

1. The extruded alloy met the specified requirement for microstructural parameters of the program. The median particle diameter was 500A° (10<sup>-10</sup>m) and 182A° (10<sup>-10</sup>m) for replica and extracted particle micrographs.
2. The best stress rupture results were obtained on the extruded alloy swaged at 982°C to 60% and 70% R.A. and heat treated for two hours at 1371°C. The 1094°C stress rupture lives were 7.2 hours at 10 KSI (69 MN/m<sup>2</sup>) and 19.8 hours at 8 KSI (55.2 MN/m<sup>2</sup>).
3. Swaging of the extruded alloy at a higher temperature of 1205°C resulted in lower stress rupture properties. The best stress rupture life obtained was 3.8 hours at 8.0 KSI (55.2 MN/m<sup>2</sup>) for the alloy swaged at 1205°C to 80% R.A. and heat treated for 2 hours at 1371°C.
4. Heat treatment of the extruded alloy for 2 hours at 1316°C prior to swaging did not yield better results. The best stress rupture life obtained was 3.3 hours at 8.0 KSI (55.2 MN/m<sup>2</sup>) for the alloy swaged at 1205°C to 80% R.A. and heat treated for 2 hours at 1371°C.
5. Lineal analysis indicated a definite trend towards an increase in the average ThO<sub>2</sub> particle diameter with processing. The average size for the extruded bar was 1132A° (10<sup>-10</sup>m). After swaging to 60% R.A. this increased to 1796A° (10<sup>-10</sup>m). And when the 60% R.A. swaged material was annealed for two hours at 1371°C, the average thoria size was 2276A° (10<sup>-10</sup>m). However, the areal analysis results showed that the median thoria particle size of the swaged and heat treated alloy was the same as that of the extruded alloy, namely 250 to 500A° (10<sup>-10</sup>m). It was therefore concluded that the swaged and heat treated alloy had sufficiently fine thoria particles (more than 50% - 250 to 500A° (10<sup>-10</sup>m) to be effective for dispersion strengthening of the alloy at elevated temperature.
6. The swaged and heat treated rods having fine elongated grains (ASTM 5 to 10) gave lower stress rupture values. The swaged and heat treated rods with the best stress rupture results had partially recrystallized duplexed grain structure consisting of a mixture of fine (ASTM 5 to 7) and coarse ASTM (1 to 3) elongated grains. Therefore it can be postulated that higher stress rupture strengths were not achieved because the alloy did not recrystallize to a uniform coarse grained structure.

7. It is evident from the foregoing conclusions that the swaging processing followed in the program was not successful in producing a high strength alloy. This probably could be attributed to inherent inability of the swaging process to impart the high strain condition or severe deformation necessary to enable the alloy to completely recrystallize to a uniform coarse grained structure.

#### Task II

1. The results of the lineal and areal analysis show that the extruded rod in Task II had a much coarser  $\text{ThO}_2$  particle size, the median  $\text{ThO}_2$  particle size being  $1250\text{\AA}$  ( $10^{-10}\text{m}$ ) as compared to  $500\text{\AA}$  ( $10^{-10}\text{m}$ ) for extruded rod in Task I.

2. The stress rupture results obtained in Task II were substantially lower than those obtained in Task I. The best stress rupture results were obtained on the extruded rod swaged at  $982^\circ\text{C}$  and heat treated for 10.5 hours at  $1205^\circ\text{C}$ . The  $1094^\circ\text{C}$  stress rupture life was 44.3 hours at 5 KSI ( $34.5\text{ MN/m}^2$ ). The median  $\text{ThO}_2$  particle size for this product was  $1000\text{\AA}$  ( $10^{-10}\text{m}$ ) and the microstructure showed fine elongated grains with uneven distribution of  $\text{ThO}_2$  particles.

3. It was concluded that the large thoria particle size and nonhomogeneous distribution were responsible for the low stress rupture strength.

4. The processing conditions such as reducing, pressing, sintering, extruding and swaging for Task I and II were identical yet the properties in Task II were considerably lower than in Task I. The main noticeable difference was in the structural characteristics of the two materials. In Task I material, the electronmicrographs showed a homogeneous dispersion of fine  $\text{ThO}_2$  particles while for Task II material, the  $\text{ThO}_2$  particles were large and unevenly dispersed. It is possible that the latter condition could have occurred during the mixing of the metal salt and colloidal  $\text{ThO}_2$ . In the process of mixing the colloidal  $\text{ThO}_2$  suspension, with the 35% nitrate salt solution, the  $\text{ThO}_2$  suspension might have become unstable and precipitated out of the nitrate solution as large thoria particles.



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TABLE 1

SCREEN ANALYSIS OF FLASH DRIED MATERIAL  
Co-18Cr-20Ni-4.6ThO<sub>2</sub> - PP3-2A

<u>U.S.S. SCREEN</u> <u>SIZE</u>	<u>WT. %</u>
+4	1.96
-4 +8	7.04
-8 +16	14.63
-16 +30	16.23
-30 +50	16.73
-50 +100	12.41
-100 +200	10.75
-200 +400	8.14
-400	12.0

TABLE 2

RESULTS OF CHEMICAL ANALYSIS OF REDUCING  
FLASH DRIED MATERIAL Co-18Cr-20Ni-4.6ThO<sub>2</sub>-  
PP3-2A UNDER CRACKED AMMONIA FOR TWO HOURS

BATCH NO.	Co Wt. %	Ni Wt. %	Cr Wt. %	ThO <sub>2</sub> Wt. %	O <sub>2</sub> Wt. %	Fe Wt. %
2	57.0	18.11	16.30	4.78	3.45	0.26
8	55.3	19.43	16.99	4.66	3.25	0.30

TABLE 3SCREEN ANALYSIS OF CALCINED MATERIALCo-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-3A

U.S.S. SCREEN SIZE	AS CALCINED (WT. %)	CALCINED & GROUND (WT. %)
+ 4	1.26	
-4 + 8	4.86	
-8 +16	10.06	
-16 + 30	14.13	
-30 + 50	16.35	1.08
-50 +100	15.23	11.05
-100 +200	13.32	27.61
-200 +400	9.90	25.94
-400	14.88	34.32

TABLE 4

SCREEN AND CHEMICAL ANALYSIS OF FIRST  
REDUCED MATERIAL Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-7A

<u>U.S.S. Screen</u>		<u>Wt. %</u>
<u>Size</u>		
+ 50		-
-50	+100	1.41
-100	+200	22.83
-200	+400	44.47
-400		31.29

Weight loss in high purity hydrogen -wt. %		8.76, 8.83
Oxygen Analysis	-wt. %	7.55, 7.65, 7.72, 7.80
ThO <sub>2</sub> Analysis	-wt. %	4.22, 4.28, 4.37, 4.48

TABLE 5CHEMICAL ANALYSIS OF POWDERS OF TWO LOT NUMBERS

<u>POWDER LOT NO.</u> <u>ELEMENT</u>	<u>SPECIFICATION</u>	<u>PP3-17A-69</u>	<u>PP3-13A-76</u>
Co	Balance	56.70 wt. %	
Cr	17.0 - 19.0 wt. %	18.25 wt. %	
Ni	19.0 - 21.0 wt. %	19.84 wt. %	
ThO <sub>2</sub>	4.4 - 4.8 wt. %	4.53 wt. %	
C	- - -	0.075 wt. %	0.043 wt. %
H	- - -	0.0017 wt. %	-
O <sup>x</sup>	- - -	0.00 wt. %	0.07 wt. %
N	- - -	0.0089 wt. %	0.0035 wt. %
P	- - -	0.0004 wt. %	
Al		0.02 wt. %	
Fe		0.44 wt. %	
Mg		0.01 wt. %	
Mn		0.01 wt. %	
Si		0.01 wt. %	
Ag, As, B, Ba, Bi, Ca, Cb, Cd, Cu, V, Li, Mg, Mn, Mo, Na, Pb, Sb, Si, Sn, Ti, V, W, Zn.		} Not Detected	

X = Non-ThO<sub>2</sub> Oxygen

TABLE 6

RESULTS OF PRESSING AND SINTERING EXPERIMENTS ON Co-18Cr-20Ni-4.6ThO<sub>2</sub> POWDERS

Sintering Run No.	45	46	47	48	50	51	52	53
Billet No.	1	2	3	4	5	6	7	8
Powder Lot No.	17A-69	17A-69	17A-69	17A-69	13A-76	13A-76	13A-76	13A-76
Pressed at MM/m <sup>2</sup>	104	104	104	104	104	104	104	104
Green Density - % Theoretical Density	55.9	57.9	56.0	56.0	55.3	55.6	55.5	56.2
Sintering Temp - °C	1205	1205	1205	1205	1205	1205	1205	1205
Sintering Times - Hours	4	4	4	4	4	4	4	4
Sintered Density - % Theoretical Density	58.0	57.9	58.3	58.1	57.4	58.3	58.1	59.1

TABLE 7

EXTRUSION VARIABLES IN THE FABRICATION OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>PP3

Can Material                      Mild Steel

Press Capacity                     $12.6 \times 10^6 \text{ N}$

Liner                                9 cm

Die                                  2.84 cm

Temperature                       $1094^\circ\text{C}$

Reduction                         10:1

Speed                               889 cms per minute

Billet No.	Extrusion No.	Lot No.	Extruding Force (N)		Extrusion Constant *K (MN/m <sup>2</sup> )	
			Upset	Running	Upset	Running
1 2	1	17A-69 17A-69	$69.75 \times 10^5$	$65.25 \times 10^5$	47.06	44.16
3 4	2	17A-69 17A-69	$72 \times 10^5$	$65.25 \times 10^5$	48.58	44.16
5 6	6	13A-76 13A-76	$67.5 \times 10^5$	$56.25 \times 10^5$	45.54	37.95
7 8	8	13A-76 13A-76	$65.25 \times 10^5$	$54 \times 10^5$	44.16	37.12

\*These data were obtained from  $K = \frac{\text{Max force to extrude tons/sq.cm}}{\text{In reduction ratio}}$



TABLE 8

EVALUATION OF EXTRUDED RODS OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3

Extrusion No.	1	2	6	8
Powder Lot No.	17A-69	17A-69	18A-76	13A-76
Carbon - wt. %	0.12	0.01	0.03	-
Non-ThO <sub>2</sub> O <sub>2</sub> - wt. %	0.01	0.04	0.01	-
Hardness - Rc	Front	38.0	38.0	38.0
	Middle	39.0	37.0	37.0
	Back	39.0	37.0	38.0
Density - % Theoretical Density	Front	99.0	99.3	99.3
	Middle	99.2	99.4	99.3
	Back	99.5	99.4	99.3
Particle Volume Fraction - Vol. %	Front	-	2.95	
	Middle	-	4.08	7.00
	Back	-	9.41	
Average Particle Diameter - A <sup>o</sup> (10 <sup>-10</sup> m)	Front	828		
	Middle	1174	1833	
	Back	1372		
Mean Free Path - Microns (μ m)	Front	1.82		
	Middle	1.82	1.60	
	Back	0.89		
Room Temperature Ultimate Tensile Strength - MN/m <sup>2</sup>	1102.62			
Yield Strength - MN/m <sup>2</sup>	1042.59			
	694.14			
	697.59			
Elongation - %	19.7			
	6.7			
Reduction in Area - %	17.8			
	9.2			
1094 <sup>o</sup> C Ultimate Tensile Strength - MN/m <sup>2</sup>	40.71			
Yield Strength MN/m <sup>2</sup>	37.26			
	28.98			
	23.46			
Elongation - %	10.7			
	19.6			
Reduction in Area %	15.1			
	16.9			

TABLE 9

RESULTS ON VARIOUS TESTS ON AS EXTRUDED AND HEAT TREATED ROD  
OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

<u>Heat Treatment</u>		Density in % Theoretical <u>Density</u>	Hardness <u>Rc</u>	<u>1094°C tensile</u>			
				<u>Y.S. MN/m<sup>2</sup></u>	<u>U.T.S.-MN/m<sup>2</sup></u>	<u>EL-%</u>	<u>R.A.-%</u>
1.	As Extruded	99.7	37	28.98	40.50	10.7	15.1
2.	1 Hr. @ 982°C	99.7	39				
3.	2 Hr. @ 982°C	99.6	37	---	28.91	16.1	15.1
4.	1 Hr. @ 1094°C	99.8	38				
5.	2 Hr. @ 1094°C	99.8	38				
6.	1 Hr. @ 1205°C	99.8	38				
7.	2 Hr. @ 1205°C	99.8	38				
8.	1 Hr. @ 1316°C	99.7	37				
9.	2 Hr. @ 1316°C	99.8	36.5	28.15	60.72	15.5	14.1
10.	4 Hr. @ 1316°C	99.6	36	28.43	33.53	8.9	8.9
11.	1 Hr. @ 1371°C	99.7	37.5				
12.	2 Hr. @ 1371°C	99.9	36.5	64.17	69.69	12.1	9.6
13.	4 Hr. @ 1371°C	99.6	35.5	83.49	86.94	13.8	13.8
14.	8 Hr. @ 1371°C	99.7	33.5	58.99	66.93	9.6	8.8
15.	16 Hr. @ 1371°C	99.5	29	74.52	80.04	8.3	5.3

TABLE 10

RESULTS OF 1094°C STRESS-RUPTURE TESTS ON AS EXTRUDED HEAT TREATED ROD  
OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

<u>Heat Treatment</u>	<u>Stress - MN/m<sup>2</sup></u>	<u>Hours</u>	<u>EL.%</u>	<u>R.A.-%</u>
2 Hours @ 1371°C	34.50	3.1	3.6	0.0
4 Hours @ 1371°C	34.50	64.5 →	---	---
	41.40	24.0 →	---	---
	48.30	24.0 →	---	---
	55.20	2.6	4.6	1.7
8 Hours @ 1371°C	34.50	24.5 →	---	---
	41.40	24.4 →	---	---
	48.30	24.0 →	---	---
	55.20	1.8	4.9	1.7
16 Hours @ 1371°C	34.50	24.0 →	---	---
	41.40	24.0 →	---	---
	48.30	24.0 →	---	---
	55.20	0.6	7.4	4.0

TABLE 11SWAGING PLAN

<u>Step No.</u>	<u>Die Size Cm</u>	<u>Cumulative % R.A.</u>	<u>Piece Size Length-Cm</u>	<u>Piece Size Length-Cm</u>
0	2.01*	---	20.32	20.32
1	1.91			
2	1.82	20	25.40	<u>25.40</u>
3	1.74			17.53
4	1.63			
5	1.55	40	33.78	<u>24.38</u>
6	1.47			16.26
7	1.39			
8	1.33			
9	1.27	60	<u>50.80</u>	<u>24.38</u>
10	1.21		25.40	11.68
11	1.15			
12	1.09	70	<u>33.53</u>	
13	1.03		20.83	
14	0.99			
15	0.95			
16	0.87	80	<u>33.02</u>	
17	0.79		20.32	
18	0.71			
19	0.64	90	<u>37.85</u>	
			25.15	

\* Bar Size

TABLE 12

TENSILE PROPERTIES OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69  
EXTRUDED 10:1 AT 1094°C AND SWAGED AT 982°C

<u>% R.A.</u>	<u>Density % of Theoretical Density</u>	<u>Hardness Rc</u>	<u>1094°C tensile</u>			
			<u>Y.S. MN/m<sup>2</sup></u>	<u>U.T.S. MN/m<sup>2</sup></u>	<u>Elong %</u>	<u>R.A. %</u>
20	99.4	39.3	17.94	21.39	11.5	18.1
40	99.6	38.7	65.55	67.62	1.9	2.3
60	99.6	40.7	76.59	77.97	1.6	2.8
70	100.4	39.3	*	81.42	0	1.8
80	99.5	39.0	77.97	81.42	3.3	5.2
90	99.4	37.7	73.83	77.28	3.3	1.8

\*Unable to find Y.S. on gauge data.

TABLE 13

TENSILE PROPERTIES OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69  
EXTRUDED 10:1 AT 1094°C AND SWAGED AT 1205°C

R.A. %	Density % of Theoretical Density	Hardness Rc	1094°C tensile test			R.A. %
			Y.S. MN/m <sup>2</sup>	U.T.S. MN/m <sup>2</sup>	Elong %	
20	99.3	39.0	28.29	47.61	16.0	11.5
40	99.1	38.0	26.91	31.05	14.8	10.7
	99.1	38.0	35.19	37.95	11.9	9.1
60	99.1	37.5	48.30	53.13	10.3	10.9
70	101.2	34.5	53.82	55.20	5.4	9.8
80	99.4	33.7	68.31	69.69	7.6	5.8
90	99.4	34.5	53.82	55.89	5.0	1.2

TABLE 14

TENSILE PROPERTIES OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69  
EXTRUDED 10:1 AT 1094°C - HEAT TREATED 2 HOURS AT 1316°C  
AND SWAGED AT 982°C.

<u>R.A.</u> <u>%</u>	<u>Density % of</u> <u>Theoretical</u> <u>Density</u>	<u>Hardness</u> <u>Rc</u>	<u>1094°C tensile test</u>			<u>R.A.</u> <u>%</u>
			<u>Y.S.</u> <u>MN/m<sup>2</sup></u>	<u>U.T.S.</u> <u>MN/m<sup>2</sup></u>	<u>Elong</u> <u>%</u>	
20	99.5	37.8	32.43	36.57	3.2	0
40	99.6	35.3	60.03	62.79	16.1	7.6
60	99.0	36.5	67.62	72.45	6.0	1.6
	99.0	36.5	53.82	61.41	4.4	0
70	99.3	36.0	111.09	115.23	4.8	0
80	99.4	36.7	66.24	67.62	1.8	0
90	99.4	36.7	48.99	50.37	3.1	1.6

TABLE 15

TENSILE PROPERTIES Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69  
 EXTRUDED 10:1 AT 1094°C - HEAT TREATED 2 HOURS AT 1316°C  
 AND SWAGED AT 1205°C

R.A. %	Density % of Theoretical Density	Hardness Rc	1094°C tensile test		Elong %	R.A. %
			Y.S. MN/m <sup>2</sup>	U.T.S. MN/m <sup>2</sup>		
20	99.6	36.0	26.98	28.84	11.1	7.8
40	99.3	36.2	32.29	35.05	7.5	4.5
60	99.1	35.7	43.13	46.58	9.5	6.7
	99.1	35.7	37.67	43.61	8.8	5.1
70	99.4	34.2	95.08	97.43	1.6	1.6
	99.4	34.2	60.79	64.31	1.8	2.8
80	99.2	34.0	60.65	66.59	4.7	4.0
90	99.3	32.5	82.46	83.21	6.6	1.7
			64.65	67.41	3.6	0.0



TABLE 16

COMPARISON OF 1094°C ULTIMATE TENSILE PROPERTIES (in MN/m<sup>2</sup>) on  
 VARIOUS THERMOMECHANICALLY WORKED SAMPLES OF  
 EXTRUDED Co-18Cr-20Ni-4.6THO<sub>2</sub>-PP3-17A-69

Bar Condition	As Swaged to % R.A.				
	0	60	70	80	90
Swaged @ 982°C	40.71	77.97	81.42	81.42	77.28
Swaged @ 1205°C	40.71	53.13	55.20	69.69	55.89
H.T.* and Swaged at 982°C	60.72	66.93	115.23	67.62	50.37
H.T.* and Swaged at 1205°C	60.72	44.85	80.73	66.93	75.21
<u>H.T.** After Swaging</u>					
Swaged @ 982°C	69.69	97.98	----	----	55.89
Swaged @ 1205°C	69.69	40.50	----	----	75.90
H.T.* and Swaged at 982°C	----	95.22	102.81	----	44.85
H.T.* and Swaged at 1205°C	----	44.85	----	----	60.03

\* Heat Treated @ 1316°C for 2 hours

\*\* Heat Treated @ 1371°C for 2 hours

TABLE 17

COMPARISON OF 1094°C, STRESS RUPTURE PROPERTIES ON  
VARIOUS THERMOMECHANICALLY WORKED EXTRUDED SAMPLES  
OF Co-18Cr-20Ni-4.6THO<sub>2</sub>-PP3-17A-69

Bar Condition	As Swaged to % R.A.							
	60		70		80		90	
	Load*	Hr.	Load*	Hr.	Load*	Hr.	Load*	Hr.
Swaged @ 982°C	48.30	0.8	----	---	48.30	23.5	41.40	6.8
Swaged @ 1205°C	34.50	0.3	----	---	41.40	0.6	34.50	6.5
H.T.** and Swaged @ 982°C	34.50	0.3	----	---	34.50	1.0	41.40	0.1
H.T.** and Swaged @ 1205°C	34.50	0.8	----	---	34.50	17.8	34.50	1.0
<u>H.T.** After Swaging</u>								
Swaged @ 982°C	69.00	7.2	55.20	19.8	----	---	55.20	0.1
	55.20	7.8						
Swaged @ 1205°C	34.50	3.7	55.20	0.1	55.20	3.8	34.50	0.1
H.T.** and Swaged @ 982°C	55.20	0.2	48.30	0.7	48.30	0.1	34.50	0.1
	34.50	3.8						
H.T.** and Swaged @ 1205°C	34.50	1.5	55.20	0.7	55.20	3.3	55.20	2.2
							41.40	1.9

\* MN/m<sup>2</sup>

\*\* Heat Treated @ 1316°C for 2 hours

\*\*\* Heat Treated @ 1371°C for 2 hours

TABLE 18

COMPARISON OF GRAIN SIZES AND GRAIN CONFIGURATIONS OF  
VARIOUS THERMOMECHANICALLY WORKED EXTRUDED  
SAMPLES OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

(Grain Sizes Given in ASTM Nos.; All Fine Grain Size ASTM 9 or finer)				
Bar Condition	As Swaged to % R.A.			
	60	70	80	90
Swaged @ 982°C	Fine Elongated	Fine Elongated	Fine Elongated	No Grains Seen
Swaged @ 1205°C	Fine Elongated	Fine Elongated	Fine Elongated	Fine Equiaxed
H.T.* and Swaged @ 982°C	Fine Elongated	Fine & coarse, Elong. & Equiaxed	Fine Elongated & Equiaxed	Fine Elongated & Equiaxed
H.T.* and Swaged @ 1205°C	60% Elongated, 5-6 40% Elongated, 9-10	Fine Elongated, 7 to 10	Fine Elongated, 9 to 10	60% Fine Elongated, 40% coarse, 5 to 6
<u>H.T.** After Swaging</u>				
Swaged @ 982°C	Large Elongated, 1-2 10% Elongated, 6-7	Elongated, 5-6	Equiaxed, 5-8 10% Elongated, 2-3	Fine to Coarse Equiaxed, 4-9
Swaged @ 1205°C	Elongated, 5 to 10	60% Elongated, 1-3, 40% Elongated, 8-9	Coarse Elongated 1-3, Equiaxed, 1-3	Fine Equiaxed
H.T.* and Swaged @ 982°C	50% Elongated, 1-2, 50% Elongated, 5-7	Mixed Elongated & Equiaxed, 3 to 9	80% Equiaxed, 5-7 20% Elongated, 5-7	Equiaxed, 7 to 9
H.T.* and Swaged @ 1205°C	Fine Elongated	30% Elongated, 1-5, 70% Elongated, 8-9	50% Elongated, 1-3, 50% Elongated, 6-7	Equiaxed, 5-8

\* Heat Treated @ 1316°C for 2 hours

\*\* Heat Treated @ 1371°C for 2 hours

TABLE 19

COMPARISON OF 1094°C TENSILE PROPERTIES OF  
 SWAGED AT 982°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Swaged to % R.A.	Heat Treatment	Heat Treat Run #	Density in %		1094°C Tensile			
			Theoretical Density	Hardness Rc	Y.S. (MN/m <sup>2</sup> )	U.T.S. MN/m <sup>2</sup> )	Elong %	R.A. %
0	----	----	99.7	37	28.98	40.71	10.7	15.1
20	----	----	99.4	39	17.94	21.39	11.5	18.1
40	----	----	99.6	39	65.55	67.62	1.9	2.3
60	----	----	99.6	41	76.59	77.97	1.6	2.8
60 2 Hr.	@ 1316°C	1	99.5	31	69.00	71.07	3.0	2.7
60 2 Hr.	@ 1371°C	2	99.8	30	93.15	97.98	4.1	3.4
70	----	----	100.4	39	*	81.42	0.0	1.8
80	----	----	99.5	39	77.97	81.42	3.3	5.2
90	----	----	99.4	38	73.83	77.28	3.3	1.8
90 2 Hr.	@ 1371°C	29	99.3	26	40.71	55.89	3.1	1.1

\*Unable to find Y.S. on gauge data

TABLE 20

COMPARISON OF 1094°C, STRESS-RUPTURE PROPERTIES OF  
SWAGED AT 982°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Bar Condition			Density in % Theoretical Density	Hardness Rc	Load (MN/m <sup>2</sup> )	Time Hr.	Elong %	R.A. %
Swaged to % R.A.	Heat Treatment	Heat Treat Run No.						
0	----	---	99.7	37	----	---	---	---
60	----	---	99.6	41	34.50 41.40 48.30	48.8 → 24.0 → 0.8	4.9	0.6
60	2 Hr. @ 1371°C	5	99.7	31	34.50 41.40 48.30 55.20 62.10 69.00	24.0 → 24.0 → 24.0 → 24.0 → 24.0 → 7.2	7.8	4.5
60	2 Hr. @ 1371°C	10	99.3	33	34.50 48.30 55.20	24.0 → 24.0 → 7.8	7.9	4.7
70	2 Hr. @ 1316°C	18	99.5	28	34.50 48.30	24.0 → 11.7	7.6	7.3
70	2 Hr. @ 1371°C	6	99.5	28	48.30 55.20	24.0 → 19.8	3.3	0.6
80	----	---	99.5	39	34.50 41.40 48.30	24.0 → 24.0 → 23.5	3.2	0.0
80	2 Hr. @ 1316°C	19	99.4	33	55.20	30 Seconds	1.4	0.5
90	----	---	99.4	37	34.50 41.40	24.0 → 6.8	3.6	1.2
90	2 Hr. @ 1316°C	20	99.4	36	55.20	0.1	2.9	1.1
90	2 Hr. @ 1371°C	21	99.4	27	55.20	0.1	---	---

TABLE 21

COMPARISON OF GRAIN SIZES AND GRAIN CONFIGURATION OF  
SWAGED AT 982°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Bar Condition Swaged to % R.A.	Heat Treatment	Heat Treat Run #	Density in % Theoretical Density	Hardness Rc	Microstructure*
0	----	--	99.7	37	Very fine equiaxed grains.
60	----	--	99.6	41	Very fine elongated grains.
60	2 Hr. @ 1316°C	1	99.5	31	Mixture of coarse (ASTM 4 to 5), and fine (ASTM 7 to 9), elongated grains.
60	2 Hr. @ 1371°C	2	99.8	30	Large elongated grains, (ASTM 1 to 2 and larger), plus scattering of fine elongated grains, (estimated 10%), (ASTM 6 to 7).
60	2 Hr. @ 1371°C	10	99.3	33	Narrow band of elongated grains, (ASTM 6 to 8) surround by coarse elongated grains, (ASTM 1 to 4).
70	----		100.4	39	Very fine elongated grains.
70	2 Hr. @ 1316°C	18	99.5	28	Very fine elongated grains.
70	2 Hr. @ 1371°C	6	99.5	28	Elongated grains, (ASTM 5 to 6)
80	----	--	99.5	39	Very fine elongated grains.
80	2 Hr. @ 1316°C	19	99.4	33	Estimated 90% fine and equiaxed grains, (ASTM 5 to 8), and coarse (ASTM 2 to 3) elongated.
90	----	--	99.4	38	No grains seen.
90	2 Hr. @ 1316°C	20	99.4	36	Fine equiaxed structure (ASTM 6 to 10).
90	2 Hr. @ 1371°C	21	99.4	27	Mixture of fine to coarse equiaxed grains, (ASTM 4 to 9).

\*Very fine grains are ASTM 9 or finer.

TABLE 22

COMPARISON OF 1094°C TENSILE PROPERTIES OF  
 SWAGED AT 1205°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Swaged to % R.A.	Heat Treatment	Heat Treat Run #	Density in % Hardness		1094°C tensile			
			Theoretical Density	Rc	Y.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )	Elong %	R.A. %
0	----	---	99.7	37	28.98	40.71	10.7	15.1
20	----	---	99.3	39	28.29	47.61	16.0	11.5
40	----	---	99.1	38	26.91	31.05	14.8	10.7
	----	---	99.1	38	35.19	37.95	11.9	9.1
60	----	---	99.1	37	48.30	53.13	10.3	10.9
60	2 Hr. @ 1371°C	32	99.8	33	32.43	40.71	3.1	2.6
70	----	---	101.2	35	53.82	55.20	5.4	9.8
80	----	---	99.4	34	68.31	69.69	7.6	5.8
90	----	---	99.4	35	53.82	55.89	5.0	1.2
90	2 Hr. @ 1371°C	3	99.4	30	71.07	75.90	4.5	2.8

TABLE 23

COMPARISON OF 1094°C STRESS RUPTURE PROPERTIES OF  
SWAGED AT 1205°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Bar Condition		Heat Treatment	Heat Treat Run No.	Density in % Theoretical Density	Hardness Rc	Lead (MN/m <sup>2</sup> )	Time Hrs.	Elong %	R.A. %
Swaged to % R.A.									
0		----	--	99.7	37	---	---	---	---
60		----	--	99.1	38	34.50	0.3	7.9	7.8
60	2 Hr. @ 1371°C		25	99.8	34	34.50	3.7	4.6	0.6
70	2 Hr. @ 1371°C		11	99.8	34	55.20	0.1	6.5	0.6
70	4 Hr. @ 1371°C		24	99.7	25	48.30	0.2	6.7	0.2
80		----	--	99.4	34	34.50 41.40	24 → 0.6	6.8	0.0
80	2 Hr. @ 1371°C		8	99.7	28	34.50 41.40 48.30 55.20	24 → 24 → 24 → 3.8	3.4	2.9
90		----	--	99.4	35	34.50	6.6	6.8	0.0
90	2 Hr. @ 1371°C		9	99.6	27	34.50	0.1	2.9	0.6



TABLE 24

COMPARISON OF GRAIN SIZES AND GRAIN CONFIGURATION OF  
SWAGED AT 1205°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Bar Condition		Heat Treatment	Heat Treat Run #	Density in % Theoretical Density	Hardness Rc	Microstructure*
Swaged to % R.A.						
0		----	--	99.7	37	Very fine equiaxed grains.
60		----	--	99.1	38	Very fine elongated grains.
60	2 Hr. @ 1371°C		25	99.8	34	Fine elongated grains, (ASTM 5 to 10).
70		----	--	101.2	35	Very fine elongated grains, (ASTM 7 - 10).
70	2 Hr. @ 1371°C		11	99.7	34	Estimated 60% large elongated grains, (ASTM 3 to 1) and 40% Clusters of fine elongated grains, (ASTM 8 to 9).
70	4 Hr. @ 1371°C		24	99.7	25	Fine elongated grains.
80		----	--	99.4	34	Very fine elongated grains.
80	2 Hr. @ 1371°C		8	99.7	28	Coarse wide elongated grains, (ASTM 1 to 3), plus some large equiaxed grains, (ASTM 1 to 3).
90		----	--	99.4	35	Fine equiaxed grains, (ASTM 9 to 10).
90	2 Hr. @ 1371°C		3	99.5	30	Equiaxed grains. (ASTM 5 to 8).

\*Very fine grains are ASTM or finer.

TABLE 25

COMPARISON OF 1094°C TENSILE PROPERTIES OF HEAT TREATED\*,  
SWAGED AT 982°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Swaged to % R.A.	Heat Treatment	Heat Treat Run #	Density in % Theore- tical Density	Hardness Rc	1094°C Tensile			
					Y.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )	Elong %	R.A. %
0	2 Hr. @ 1316°C	4A	99.8	37	28.29	60.72	15.5	14.1
20	----	--	99.5	38	32.43	36.57	3.2	0.0
40	----	--	99.6	35	60.03	62.79	16.1	7.6
60	----	--	99.0	37	67.62	72.45	6.0	1.6
60	----	--	99.0	37	53.82	61.41	4.4	0.0
60	2 Hr. @ 1371°C	13	99.3	31	93.15	95.22	4.5	0.6
70	----	--	99.3	36	111.09	115.23	4.8	0.0
70	2 Hr. @ 1371°C	4	99.5	31	98.67	102.81	6.5	1.1
80	----	--	99.4	37	66.24	67.62	1.8	0.0
90	----	--	99.4	37	48.99	50.37	3.1	1.6
90	2 Hr. @ 1371°C	33	99.78	25	40.02	44.85	0.0	0.0

\* 2 Hours at 1316°C

TABLE 26

COMPARISON OF 1094°C STRESS RUPTURE PROPERTIES OF HEAT TREATED\*,  
SWAGED AT 982°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Bar Condition		Density in %		Hardness		Time Hrs.	Elong %	R.A. %
Swaged to % R.A.	Heat Treatment	Heat Treat Run No.	Theoretical Density	Rc	Load (MN/m <sup>2</sup> )			
0	2 Hr. @ 1316°C	4A	99.8	37	---	---	---	---
60	----	--	99.0	37	34.50	0.3	5.3	2.8
60	2 Hr. @ 1371°C	12	99.6	30	55.20	0.2	7.7	0.0
60	2 Hr. @ 1371°C	30	99.7	31	34.50	3.8	0.0	0.0
70	2 Hr. @ 1371°C	7	99.9	--	48.30	0.7	1.6	0.0
80	----	--	99.4	37	34.50	1.0	6.3	0.0
80	2 Hr. @ 1371°C	23	99.6	30	48.30	0.1	--	--
90	----	--	99.4	37	34.50 41.40	24→ 0.1		
							70	1.2
90	2 Hr. @ 1371°C	31	100.2	32	34.50	0.1	3.3	0.0

\* 2 Hours @ 1316°C.

TABLE 27

COMPARISON OF GRAIN SIZE AND GRAIN CONFIGURATION OF HEAT TREATED\*,  
SWAGED AT 982°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Bar Condition		Density in %		Microstructure**	
Swaged to	Heat Treat	Theoretical	Hardness		
% R.A.	Heat Treatment	Run No.	Density	Rc	
0	2 Hr. @ 1316°C	4A	99.8	37	Fine equiaxed grains.
60	----	--	99.0	37	Fine elongated grains.
60	2 Hr. @ 1371°C	12	99.5	30	Mixture of elongated grains, (estimated 50% ASTM 1 to 2), and 50% (ASTM 5 to 7).
70	----	--	99.3	36	Mixture of fine and coarse elongated and equiaxed grains, (ASTM 6 to 10).
70	2 Hr. @ 1371°F	4	99.5	31	Mixture of elongated and equiaxed grains, (ASTM 3 to 9).
80	----	--	99.4	37	Mixture of fine elongated and equiaxed grains, (ASTM 8 to 10).
80	2 Hr. @ 1371°C	23	99.6	30	Estimated 80% equiaxed grains, (ASTM 5 to 7), and 20% elongated grains, (ASTM 5 to 7).
90	----	--	99.4	37	Mixture of fine, (ASTM 8 to 10), equiaxed and elongated grains.
90	2 Hr. @ 1371°C	31	100.2	32	Equiaxed grains, (ASTM 7 to 9).

\* Heat Treated 2 hours @ 1316°C .

\*\* Fine and Very Fine Grains are ASTM 9 or finer.

TABLE 28

COMPARISON OF 1094°C TENSILE PROPERTIES OF HEAT TREATED,\*  
 SWAGED AT 1205°C AND HEAT TREATED SAMPLES OF  
 Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Swaged to R.A.	Heat Treatment	Heat Treat Run #	Density in % Theore- tical Density	Hardness Rc	1094°C Tensile			
					Y.S. (MN/m <sup>2</sup> )	U.T.S. (MN/m <sup>2</sup> )	Elong. %	R.A. %
0	2 Hr. @ 1316°C	4A	99.8	37	28.15	60.72	15.5	14.1
20	----	--	99.6	36	26.91	28.98	11.1	7.8
40	----	--	99.3	36	32.43	35.19	7.5	4.5
60	----	--	99.1	36	43.47	46.92	9.5	6.7
	----	--	99.1	36	37.95	43.47	8.8	5.1
60	2 Hr. @ 1371°C	28	99.8	35	37.26	44.85	9.5	5.6
70	----	--	99.4	34	95.22	97.29	1.6	1.6
	----	--	99.4	34	60.72	64.17	1.8	7.8
80	----	--	99.2	34	60.72	66.93	4.7	4.0
90	----	--	99.3	32	82.80	83.49	6.6	1.7
					64.86	67.62	3.6	0.0
90	2 Hr. @ 1371°C	17	99.5	28	55.89	60.03	4.5	2.9

\* 2 hours at 1316°C

TABLE 29

COMPARISON OF 1094°C STRESS-RUPTURE VALUES ON HEAT TREATED\*,  
SWAGED AT 1205°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Bar Condition		Heat Treat Run No.	Density in % Hardness		Load (MN/m <sup>2</sup> )	Time Hrs.	Elong %	R.A. %
Swaged to % R.A.	Heat Treatment		Theoretical Density	Rc				
0	2 Hr. @ 1316°C	4A	99.8	37	---	---	---	---
60	----	--	99.1	36	34.50	0.8	8.5	5.2
60	4 Hr. @ 1316°C	35	99.8	35	34.50	1.5	4.9	2.2
60	2 Hr. @ 1371°C	27	99.8	35	34.50	1.5	7.6	0.6
70	2 Hr. @ 1371°C	14	99.4	31	55.20	0.7	3.3	0.6
80	----	--	99.2	34	34.50	17.8	3.5	---
80	2 Hr. @ 1371°C	15	99.5	30	55.20	3.3	10.0	2.8
90	----	--	99.3	33	34.50	1.0	1.6	0.0
90	2 Hr. @ 1371°C	16	99.4	32	55.20	0.2	1.6	0.6
90	2 Hr. @ 1371°C	34	99.8	21	34.50 41.40	24 1.9	1.3	0.0

\* 2 Hours @ 1316°C

TABLE 30

COMPARISON OF GRAIN SIZE AND GRAIN CONFIGURATION OF HEAT TREATED\*,  
 SWAGED AT 1205°C AND HEAT TREATED SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Bar Condition		Heat	Density in %	Hardness	Microstructure**
Swaged to % R.A.	Heat Treatment	Treat Run #	Theoretical Density		
0	2 Hr. @ 1316°C	4A	99.8	37	Fine equiaxed grains.
60	----	--	99.1	36	Estimated 60% elongated grains, (ASTM 5 to 6), plus 40% clusters of fine elongated grains, (ASTM 9 to 10).
60	4 Hr. @ 1316°C	35	99.7	35	Fine elongated grains, (ASTM 9 to 10).
60	2 Hr. @ 1371°C	27	99.8	35	Fine elongated grains, (ASTM 9 to 10).
70	----	--	99.4	34	Elongated grains, (ASTM 7 to 10).
70	2 Hr. @ 1371°C	14	99.4	31	Mixture of fine and coarse elongated grains. Estimated 70% fine, (ASTM 8 to 9), and 30% coarse, (ASTM 1 to 5).
80	----	--	99.2	34	Fine elongated grains, (ASTM 9 to 10).
80	2 Hr. @ 1371°C	15	99.5	30	Mixture of fine and coarse elongated grains. Estimated 50% fine, (ASTM 6 to 7), and 50% coarse, (ASTM 1 to 3).
90	----	--	99.3	33	Mixture of fine and coarse elongated grains. Estimated 60% fine, (ASTM 9 to 10), and 40% coarse.
90	2 Hr. @ 1371°C	16	99.4	32	Equiaxed grains, (ASTM 5 to 8).

\* Heat Treated 2 hours @ 1316°C

\*\* Fine and Very Fine Grains are ASTM 9 or finer.

TABLE 31

SUMMARY OF LINEAL AND AREAL ANALYSIS ON Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69

Lineal Analysis				Areal Analysis		Stress Rupture Properties @ 1094°C	
Vol %	Avg Diam. A <sub>0</sub> (10 - 10 <sub>m</sub> )	Mean Free Path Micron (4m)	Median Particle Size A <sub>0</sub>	Vol %	Stress MN/m <sup>2</sup>	Hours to Rupture	
4.08	1174	1.83	500	5.46	---	---	
6.92	1132	1.04	250	7.73	55.2	2.6	
8.07	1314	0.99	250	7.29	55.2	2.6	
8.59	1796	1.27	250	8.40	48.3	0.8	
8.12	2276	1.72	250	6.32	69.1	7.2	
6.36	1832	17.94	500	4.34	---	---	
8.57	2102	1.49	500	7.35	55.2	19.8	
10.39	2202	1.27	250	4.36	34.5	0.3	

Material

As extruded - Middle Piece,  
Photo 9492

As extruded - Middle Piece, & Heat  
Treat 4 hours @ 1371°C, Photo 9494

As extruded - Middle Piece, & Heat  
Treat 4 hours @ 1371°C, Photo 9495

Swaged to 60% R.A. @ 982°C,  
Photo 9613

Swaged to 60% R.A. @ 982°C, & Heat  
Treat 2 hours @ 1371°C, Photo 9614

Swaged to 70% R.A. @ 982°C,  
Photo 9615

Swaged to 70% R.A. @ 982°C, & Heat  
Treat 2 hours @ 1371°C, Photo 9616

Swaged to 60% R.A. @ 1205°C,  
Photo 9617



TABLE 32

SWAGING OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76 BY ROUTE 1 AND 2

Swaging Step No.	Die Size CM	% Reduction in area per pass based on previous R.A.	Cumulative R.A.	Time at Temp. Min.	Route 1 Temp. °C	Route 2 Temp °C
0	2.007	0	0	60	982	982
1	1.905	11	11	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
2	1.824	8.3	20	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
3	1.745	8.1	24	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
4	1.626	13.1	34	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
5	1.547	9.5	40	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
6	1.468	10.0	46	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
7	1.387	10.8	53	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
8	1.326	8.7	58	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
9	1.270	8.1	60	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
10	1.209	9.2	64	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982
11	1.151	10.7	67	--	982	982
	---	--		15	1205	1094
	---	--		15	982	982

TABLE 33SWAGING OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76 BY ROUTE 3

Swaging Step No.	Die Size CM	% Reduction in area per pass based on previous R.A.	Cumulative % R.A.	Temp. °C	Time at Temp. Min.
0	2.007	0	0	982	60
1	1.905	11	11	982 982	-- 30
2	1.824	8.3	20	982 982	-- 30
3	1.745	8.1	24	982 982	-- 30
4	1.626	13.1	34	982 982	-- 30
5	1.547	9.5	40	982 982	-- 30
6	1.468	10.0	46	982 982	-- 30
7	1.387	10.8	53	982 982	-- 30
8	1.326	8.7	58	982 982	-- 30
9	1.270	8.1	60	982 982	-- 30
10	1.209	9.2	64	982 982	-- 30
11	1.151	10.7	67	982 982	-- 30
12	1.095	9.3	70	982	--

TABLE 34

COMPARISON OF 1094°C TENSILE PROPERTIES (MN/m<sup>2</sup>) ON  
SWAGED SAMPLES OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

Swaging Route	Density in % Theoretical Density	Hardness Rc	Y.S.	U.T.S.	Elong %	R.A. %
Route 1	99.0	38	61.41	64.17	5.0	1.0
		39	73.83	75.90	1.5	1.6
Route 2	99.6	38	66.24	69.69	4.3	2.9
		38	63.48	66.24	4.2	2.8
Route 3	99.5	40	61.41	66.24	6.2	1.7
		38	64.86	65.55	4.6	2.3

TABLE 35

COMPARISON OF 1094°C STRESS RUPTURE VALUES ON SWAGED  
SAMPLES OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

Swaging Route	Density in % Theoretical Density	Hardness Rc	Load (MN/m <sup>2</sup> )	Time Hours	Elong %	R.A. %
Route 1	99.0	38	34.50	4.5	1.4	0.0
	99.0	38	34.50	3.7	1.5	0.0
Route 2	99.6	38	34.50	2.5	1.5	0.0
	99.6	38	34.50	3.1	1.5	0.6
Route 3	99.5	40	34.50	16.7	2.9	1.1
	99.5	40	34.50	16.4	1.6	2.2

TABLE 36

COMPARISON OF GRAIN SIZE AND CONFIGURATION ON SWAGED  
SAMPLES OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

Swaging Route	Density in % Theoretical Density	Hardness Rc	Grain Size and Configuration
Route 1	98.9	38	ASTM 7 to 10; equiaxed
	99.1	39	
Route 2	99.5	38	Finer than ASTM 10; elongated
	99.5	38	
Route 3	99.5	40	Finer than ASTM 10; elongated
	99.2	38	

TABLE 37

COMPARISON OF 1094°C TENSILE PROPERTIES (MN/m<sup>2</sup>) ON SWAGED AND HEAT TREAT  
2 HOURS AT 1371°C SAMPLES OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

Swaging Route	Heat Treat #	Density in %	Hardness Rc	Y.S.	U.T.S.	Elong %	R.A. %
		Theoretical Density					
1	1B	99.6	31	51.06	51.75	1.4	0.6
		99.6		53.82	53.82	1.4	0.6
2	2B-L	99.8	28	68.31	75.21	4.8	0.5
	2B-R	99.7		59.34	60.72	0.0	0.0
3	3B-L	99.5	28	64.17	68.31	1.4	1.1
	3B-R	99.5		53.82	53.82	1.4	0.5

TABLE 38

COMPARISON OF 1094°C STRESS-RUPTURE PROPERTIES ON SWAGED AND HEAT TREAT  
2 HOURS @ 1371°C SAMPLES OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

Swaging Route	Heat Treat #	Density in %	Hardness Rc	Load (MN/m <sup>2</sup> )	Time Hrs.	Elong %	R.A. %
		Theoretical Density					
1	6B	99.5	29	34.50	0.1	---	---
	6B	99.4	29	34.50	0.1	3.1	0.0
2	7B	99.8	27	34.50	0.1	3.0	0.0
	7B	99.6		34.50	0.1	3.0	0.0
3	8B	99.6	30	34.50	1.0	2.9	0.0
	8B	99.5		34.50	0.9	1.4	0.0

TABLE 39

COMPARISON OF GRAIN SIZE AND GRAIN CONFIGURATION ON SWAGED AND HEAT TREAT  
2 HOURS AT 1371°C SAMPLES OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

Swaging Route	Heat Treat #	Density in %	Hardness Rc	Grain Size and Configuration
		Theoretical Density		
1	1B	99.3	31	20% ASTM 6-9; elongated
	6B	99.4	29	80% ASTM 6-9; equiaxed
2	2B	99.9	28	60% ASTM 4-6, 30% ASTM 6-9,
	7B	99.1	27	10% finer than ASTM 10; all elongated.
3	3B	99.3	28	70% ASTM 6-9, 30% ASTM 5-6;
	8B	99.5	30	all elongated.

TABLE 40

COMPARISON OF 1094°C TENSILE PROPERTIES (IN MN/m<sup>2</sup>) ON SWAGED AND HEAT TREAT  
4 HOURS AT 1371°C SAMPLES OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

Swaging Route	Heat Treat #	Density in % Theoretical Density	Hardness Rc	Y.S.	U.T.S.	Elong %	R.A. %
2	13B	99.7	28	55.20	55.89	3.2	0.0
3	15B	99.4	29	69.69	71.07	3.3	1.2

TABLE 41

COMPARISON OF GRAIN SIZE AND GRAIN CONFIGURATION ON SWAGED AND HEAT TREAT  
4 HOURS AT 1371°C SAMPLES OF EXTRUDED Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

Swaging Route	Heat Treat #	Density in % Theoretical Density	Hardness Rc	Grain Size and Configuration
2	13B	99.3	28	70% Finer than ASTM 10; elongated 20% ASTM 7-9; elongated 10% ASTM 5; elongated
3	15B	99.2	29	10% Finer than ASTM 10; elongated 70% ASTM 6-9; elongated 20% ASTM 5; elongated

TABLE 42

COMPARISON OF 1094°C STRESS-RUPTURE PROPERTIES ON SAMPLES OF  
 Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76 WHICH WERE SWAGED AT 982°C TO 70% R.A.  
 BY ROUTE 3 AND FURTHER HEAT TREATED AFTER SWAGING

Heat Treatment	Hardness Rc	Load MN/m <sup>2</sup>	Time Hrs.	Elong %	R.A. %
---	40	34.5	16.7	2.9	1.1
---	40	34.5	16.4	1.6	2.2
1 Hr. @ 1205°C	36	34.5	5.5	4.4	0.5
2 Hr. @ 1205°C	34	34.5	8.1	1.6	1.1
4 Hr. @ 1205°C	31	34.5	10.6	2.9	0.5
5-1/4 Hr. @ 1205°C	32	34.5	1.5	2.8	1.1
* 5-1/4 Hr. @ 1205°C	--	34.5	2.1	1.4	0.0
6-1/2 Hr. @ 1205°C	--	34.5	15.0	4.2	0.0
* 6-1/2 Hr. @ 1205°C	32	34.5	0.4	2.8	1.1
10-1/2 Hr. @ 1205°C	32	34.5	44.3	1.8	0.5
* 10-1/2 Hr. @ 1205°C	--	27.6	4.3	0.0	0.0
10-1/2 Hr. @ 1205°C	34	34.5	15.6	1.4	0.5
14.5 Hr. @ 1205°C	33	34.5	0.7	3.3	1.1
24 Hr. @ 1205°C	34	34.5	0.7	3.0	0.0
64 Hr. @ 1205°C	31	34.5	0.5	1.5	0.0
1 Hr. @ 1316°C	31	34.5	2.1	1.4	0.0
2 Hr. @ 1316°C	30	34.5	8.1	3.1	1.1
4 Hr. @ 1316°C	--	34.5	1.7	1.5	0.0
6-1/2 Hr. @ 1316°C	30	34.5	1.0	1.4	0.0

\* Heat Treated in High Purity H<sub>2</sub>. All other Samples heat treated in high purity Argon.

TABLE 43

COMPARISON OF GRAIN SIZE AND CONFIGURATION ON SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76 WHICH WERE SWAGED AT 982°C TO  
70% R.A. BY ROUTE 3 AND FURTHER HEAT TREATED AFTER SWAGING

Heat Treatment	Hardness		Grain Size and Configuration
	Rc		
---	40		100% Finer than ASTM 10; elongated
---	38		
1 Hr. @ 1205°C	36		90% Finer than ASTM 10; elongated 10% ASTM 9-10; elongated
2 Hr. @ 1205°C	34		80% Finer than ASTM 10; elongated 20% ASTM 6-8; elongated
4 Hr. @ 1205°C	31		60% Finer than ASTM 10; elongated 20% ASTM 6-9; elongated 20% ASTM 6-9; equiaxed
5-1/4 Hr. @ 1205°C	32		40% Finer than ASTM 10; elongated 30% ASTM 6-9; elongated 30% ASTM 6-9; equiaxed
* 5-1/4 Hr. @ 1205°C	--		20% Finer than ASTM 10; elongated 80% ASTM 6-10; elongated
6-1/2 Hr. @ 1205°C	--		20% Finer than ASTM 10; elongated 80% ASTM 6-8; elongated
* 6-1/2 Hr. @ 1205°C			100% Finer than ASTM 10; elongated
10-1/2 Hr. @ 1205°C			40% Finer than ASTM 10; elongated 60% ASTM 7-9; elongated
10-1/2 Hr. @ 1205°C	34		80% Finer than ASTM 10; elongated 20% ASTM 7-10; elongated
* 10-1/2 Hr. @ 1205°C	--		30% Finer than ASTM 10; elongated 35% ASTM 7-10; elongated 35% ASTM 7-10; equiaxed
14.5 Hr. @ 1205°C	33		60% Finer than ASTM 10; elongated 40% ASTM 6-10; elongated
24 Hr. @ 1205°C	34		40% Finer than ASTM 10; elongated 30% ASTM 6-10; elongated 30% ASTM 6-10; equiaxed
64 Hr. @ 1205°C	31		30% Finer than ASTM 10; elongated 30% ASTM 6-10; elongated 40% ASTM 6-10; equiaxed

\* Ran Test using Hi Purity H<sub>2</sub>.

TABLE 43

COMPARISON OF GRAIN SIZE AND GRAIN CONFIGURATION ON SAMPLES OF  
Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76 WHICH WERE SWAGED AT 982°C TO  
70% R.A. BY ROUTE 3 AND FURTHER HEAT TREATED AFTER SWAGING

<u>Heat Treatment</u>	<u>Hardness</u>	
	<u>Rc</u>	<u>Grain Size and Configuration</u>
1 Hr. @ 1316°C	31	40% Finer than ASTM 10; elongated 60% ASTM 6-10; elongated
2 Hr. @ 1316°C	30	60% ASTM 7-10; elongated 40% ASTM 5-6; elongated
4 Hr. @ 1316°C	30	10% Finer than ASTM 10; elongated 80% ASTM 6-9; elongated 10% ASTM 5; elongated
6-1/2 Hr. @ 1316°C	30	60% ASTM 5-8; elongated 40% ASTM 5-8; equiaxed



TABLE 44

SUMMARY OF LINEAL AND AREAL ANALYSIS ON Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76

	Lineal Analysis			Areal Analysis	
	Volume %	Average Diameter A <sub>O</sub> (10 <sup>-10</sup> m)	Mean Free Path Micron (μm)	Median Particle Size A <sub>O</sub> (10 <sup>-10</sup> m)	Volume %
As extruded	4.07	1823	1.60	1250	6.44
Swaged to 70% R.A., Route 3 and Heat Treat at 1205°C for 10.5 Hours	7.50	2016	1.48	1000	5.59

TABLE 45

TENSILE PROPERTIES OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76  
EXTRUDED 10:1 AT 1094°C, SWAGED TO 70% R.A. AT 982°C  
AND ANNEALED AT 982°C BETWEEN SWAGING PASSES

Test Temp °C	Y.S. MN/m <sup>2</sup>	U.T.S. MN/m <sup>2</sup>	Elong %	R.A. %
Room Temp.	848	1094	4.5	6.2
649	499	555	16.4	23.5
760	303	337	5.8	14.5
871	201	206	5.8	7.5
982	117	122	4.4	1.7
1,094	61	66	6.2	1.7
	64	66	4.6	2.3
1,191	46	48	2.8	0.5

TABLE 46

STRESS RUPTURE PROPERTIES OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76  
EXTRUDED 10:1 AT 1094°C, SWAGED TO 70% R.A. AT 982°C AND  
ANNEALED AT 982°C BETWEEN SWAGING PASSES

Test Temp °C	Load MN/m <sup>2</sup>	Time Hrs.	Elong %	R.A. %
982	88	0.5	4.3	4.0
	69	5.4	2.8	4.0
982	59	33.2	2.8	--
	59	100.9	2.9	--
982	34	2349.5 **		
	34	2159.4 **		
1,094	34	16.7	2.9	1.1
	34	16.4	1.6	2.2
1,094	21	1475	3.1	2.9
	21	1285 *	---	---

\* Test discontinued a/c machine malfunction

\*\* Both tests stopped

TABLE 47

STRESS RUPTURE PROPERTIES OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76  
EXTRUDED 10:1 AT 1094°C, SWAGED TO 70% R.A. AT 982°C AND  
ANNEALED AT 982°C BETWEEN SWAGING PASSES

	Stress in KSI (MN/m <sup>2</sup> )	
	Temperature	
	982°C	1094°C
10 hours	9.5 (65.55)	5.25 (36.23)
100 hours	8.5 (58.65)	4.0 (27.60)
1000 hours	7.0 (48.3)	3.1 (21.39)

TABLE 48

X-RAY DIFFRACTION ANALYSIS OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76,  
SWAGED AT 982°C TO 70% R.A. BY ROUTE 3

<u>Line</u>	<u>(hkl)</u>	<u>2<math>\theta</math></u> <u>Measured</u>	<u>2<math>\theta</math></u> <u>Corrected</u>	<u>d (Å)</u>	<u>a (Å)</u>
Si	(422)	38.006°	88.030°	-	-
Si	(511)	94.920°	94.952°	-	-
F.C.C.	(311)	92.100°	92.128°	1.075	3.562
Si	(311)	56.120°	56.122°	-	-
ThO <sub>2</sub>	(311)	54.500°	54.502°	1.685	5.590
Analytical Precision		±0.01°	±0.01°	-	±0.001

FIGURE 1

STEP CHART SHOWING MANUFACTURE OF ALLOY - OXIDE  
POWDERS AND CONVERSION INTO SWAGING ROD

<u>Step Number</u>	<u>Step Number</u>
1A. Dissolve	14. Evaluate Billet
1B. Filter	15. Can Billet
1C. Stock Solutions	16. Extrude to Rod
1D. Mix Solutions	17. Decan
2. Flash Dry	18. Evaluate Extruded Rod
3. Calcine	19. Heat Treat Extruded Rod
4. Grind	20. Evaluate Heat Treated Rod
5. 1st Reduce	21. Swage Extruded Rod
6. Grind	22. Evaluate Swaged Rod
7. 2nd Reduce	23. Heat Treat Swaged Rod
8. 3rd Reduce	24. Evaluate Heat Treated Swaged Rod
9. Grind	25. Swage Extruded Rod
10. Analyze	26. Heat Treat Swaged Rod
11. Blend	27. Evaluate Heat Treated Swaged Rod
12. Press	28. Select One Swaging Process
13. Sinter	29. Test Selected Swaging Process

FIGURE 2  
SINTERING APPARATUS

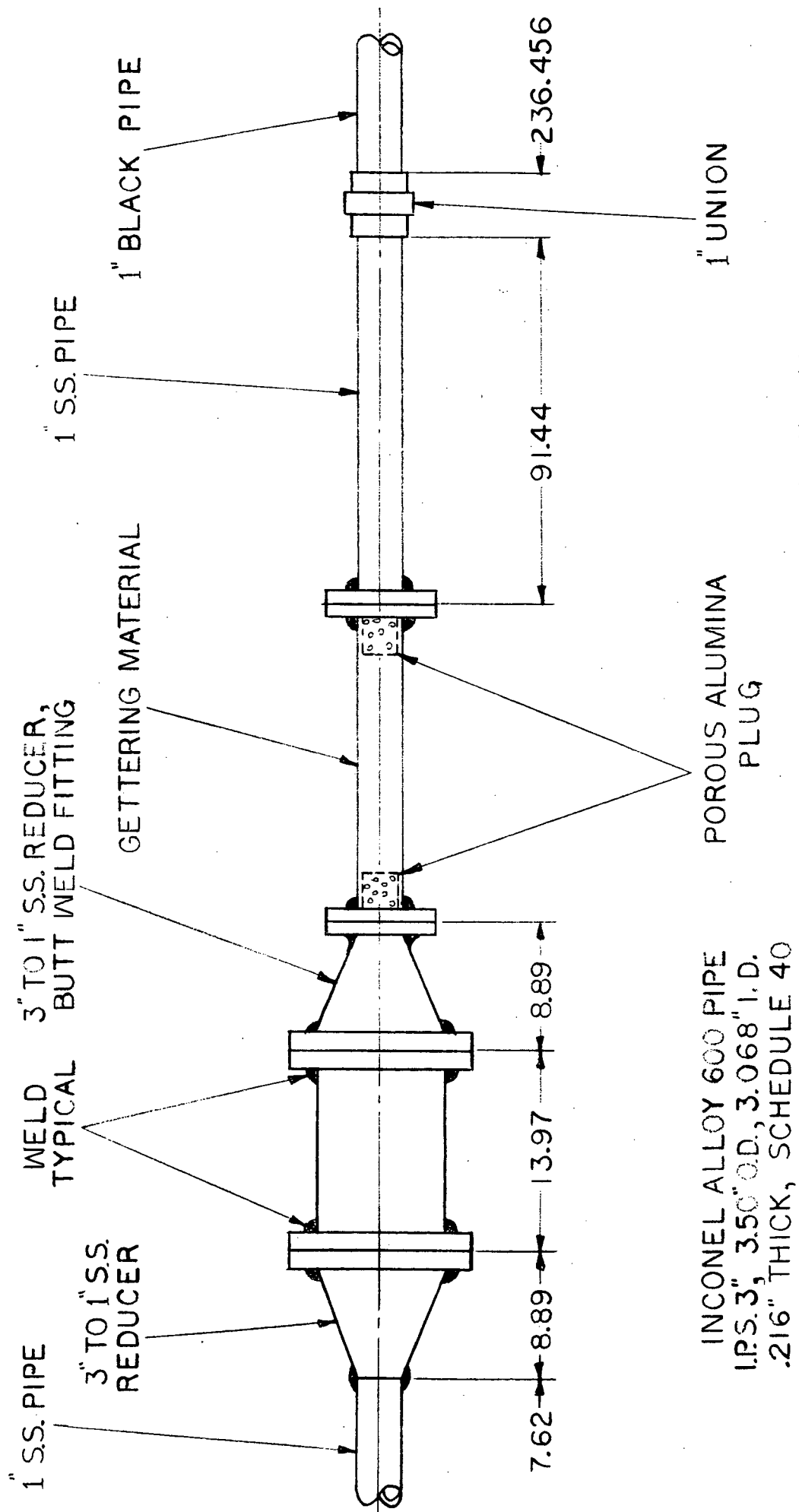
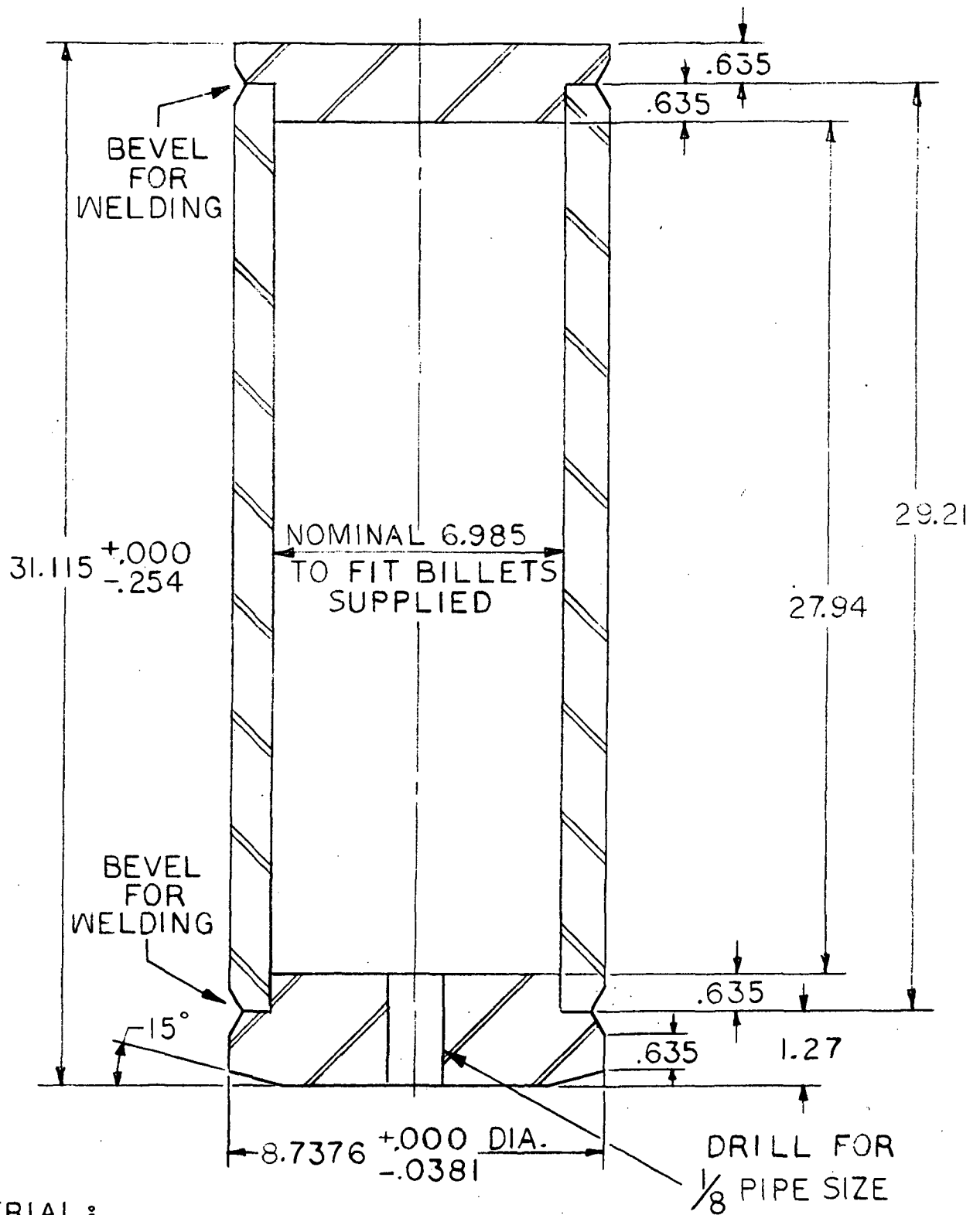


FIGURE 3

68.

CANS FOR TEST EXTRUSIONS



MATERIAL:  
LOW CARBON COLD DRAWN SEAMLESS  
MECHANICAL TUBING  
COLD ROLLED STEEL  
FOR END PLATES

ALL DIM. IN CM.



Figure 4A

Photo. No. P4634  
Extrusion #2  
Middle of Back Piece

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C

Mag. 100X  
Etchant: Electrolytic  
Perchloric Acid



Figure 4B

Photo. No. P4635  
Extrusion #2  
Middle of Back Piece

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



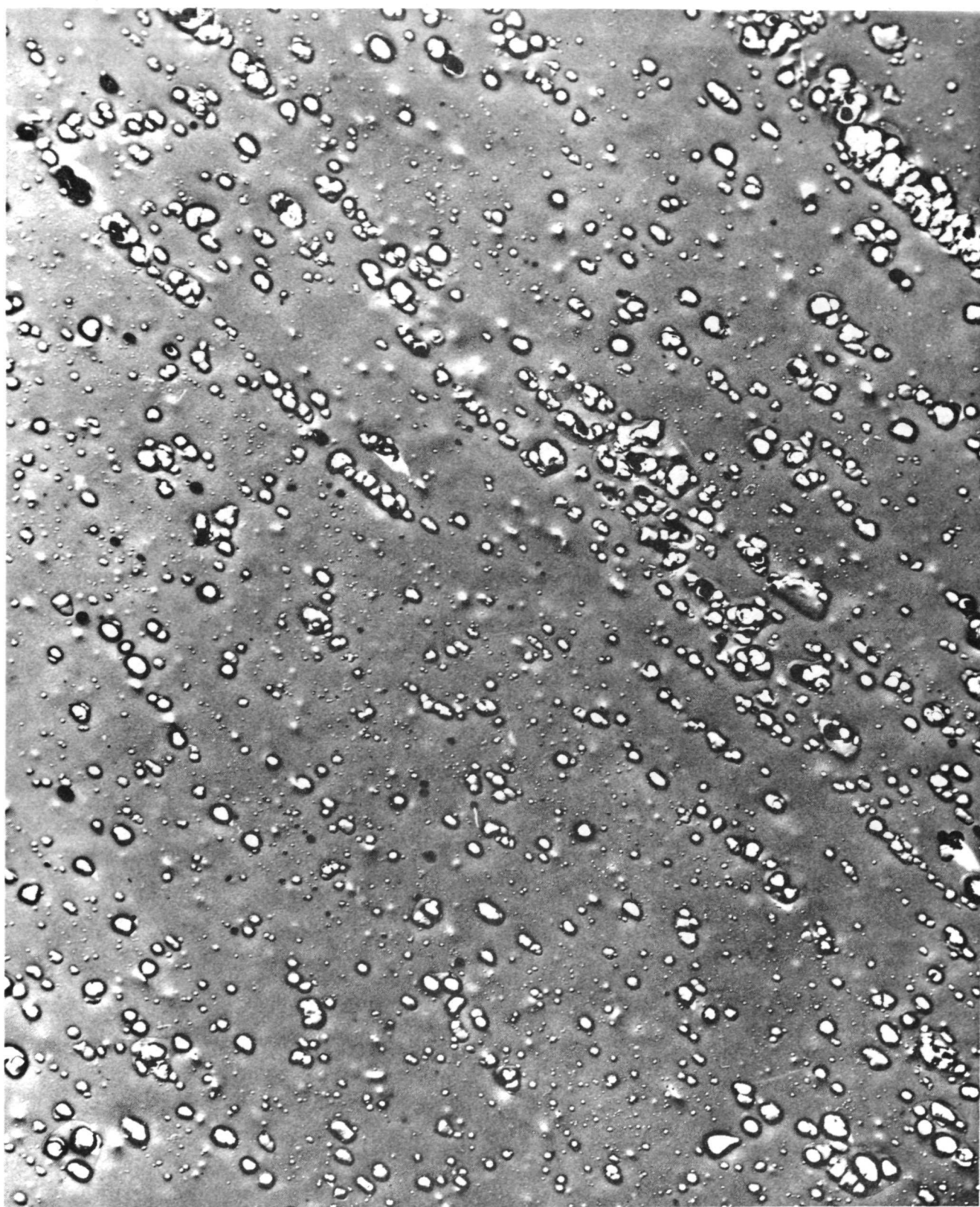


Figure 5

Photo. No. 9491  
J.R. No. 7099-3  
Extrusion #2  
Front End

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C

Mag. 10,000 X

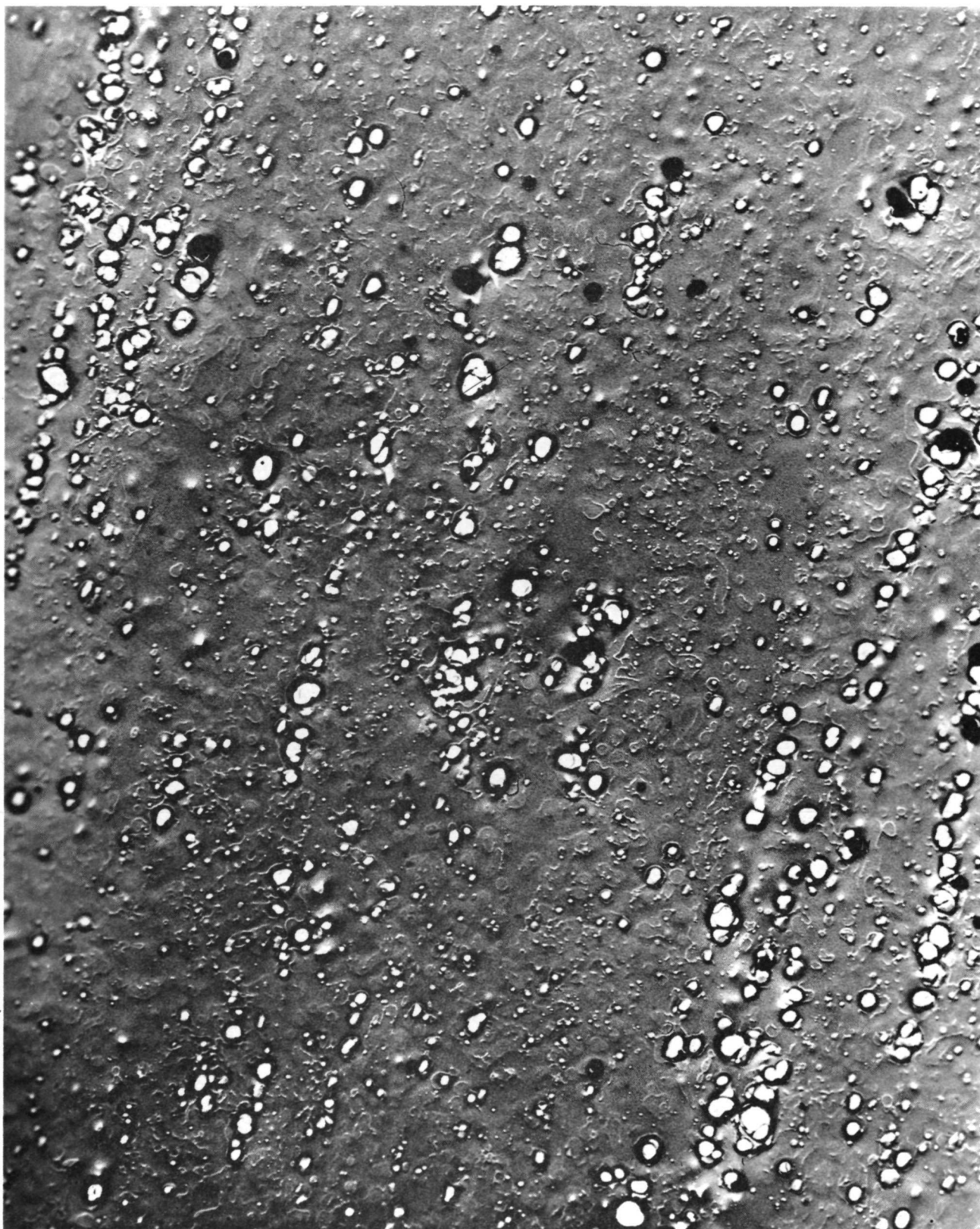


Figure 6

Photo. No. 9492  
J.R. No. 7104-1  
Extrusion #2  
Middle Section

Co-18Cr-20Ni-4.0ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C

Mag. 10,000 X



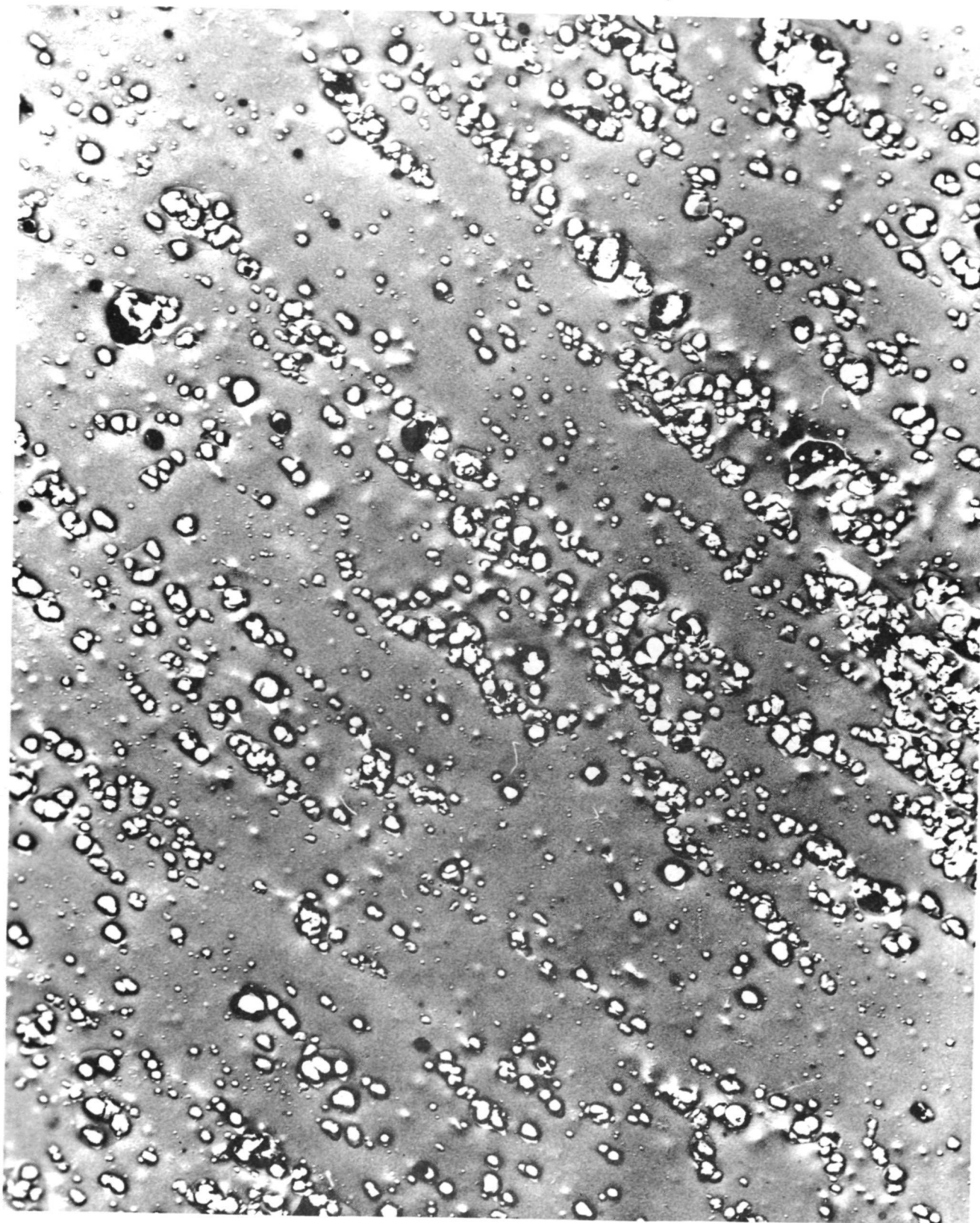


Figure 7

Photo. No. 9493  
J.R. No. 7107-3  
Extrusion #2  
Back End

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C

Mag. 10,000 X

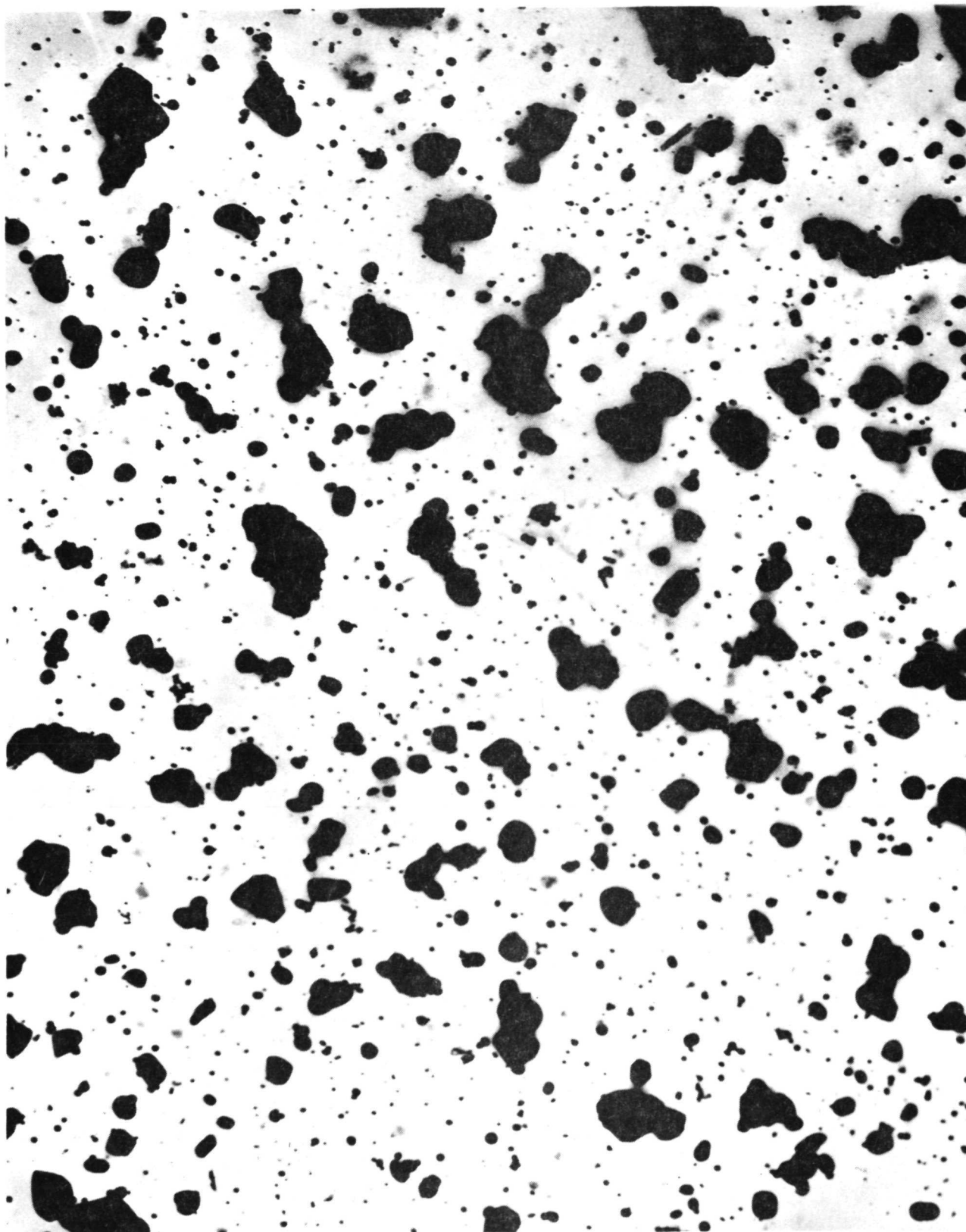


Figure 8

Photo. No. 9504  
J.R. No. 7113A  
Extrusion #2, 17A-69  
Middle Section

Extracted  $\text{ThO}_2$  particles  
Co-18Cr-20Ni-4.6 $\text{ThO}_2$ -pp3  
Extruded 10:1 @ 1094°C

Mag. 27,500 X



Figure 9A

Photo No. P4711  
PP3-13A-76  
Extrusion No. 6  
(Middle)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Density 99.33% of  
Theoretical Density

Mag. 100 x  
Etchant: Electrolytic  
Perchloric Acid

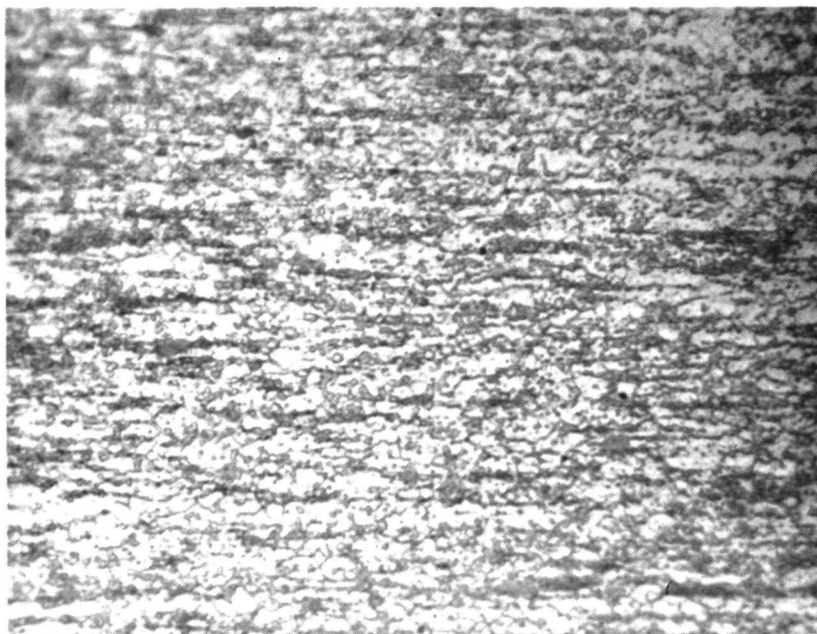


Figure 9B

Photo. No. P4712  
PP3-13A-76  
Extrusion No. 6  
(Middle)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Density 99.33% of  
Theoretical Density

Mag. 1000 x  
Etchant: Electrolytic  
Perchloric Acid





Figure 10

Photo. No. 9733  
J.R. No. 7250  
PP3-13A-76  
Extrusion #6

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C

Mag. 10,000 X

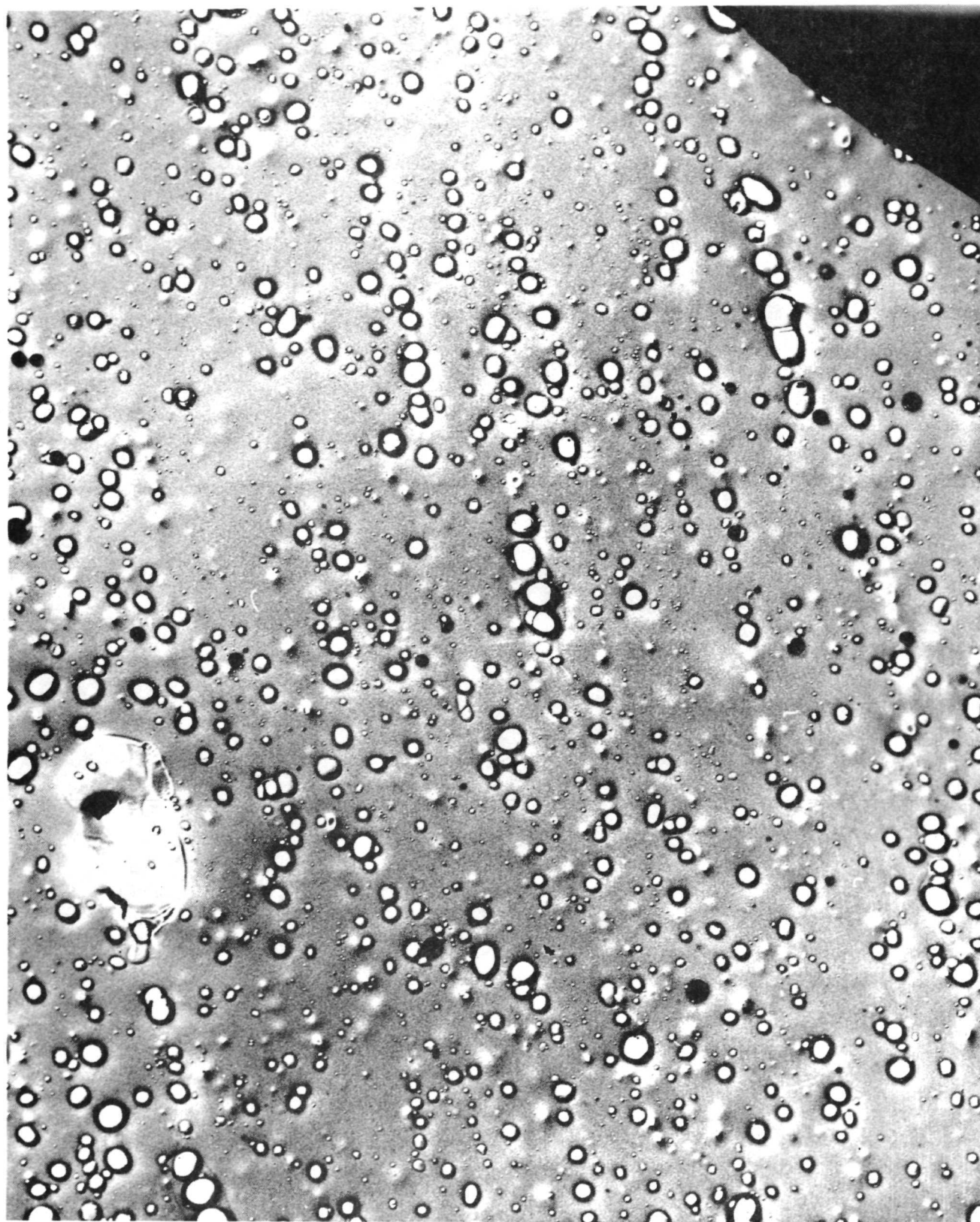


Figure 11

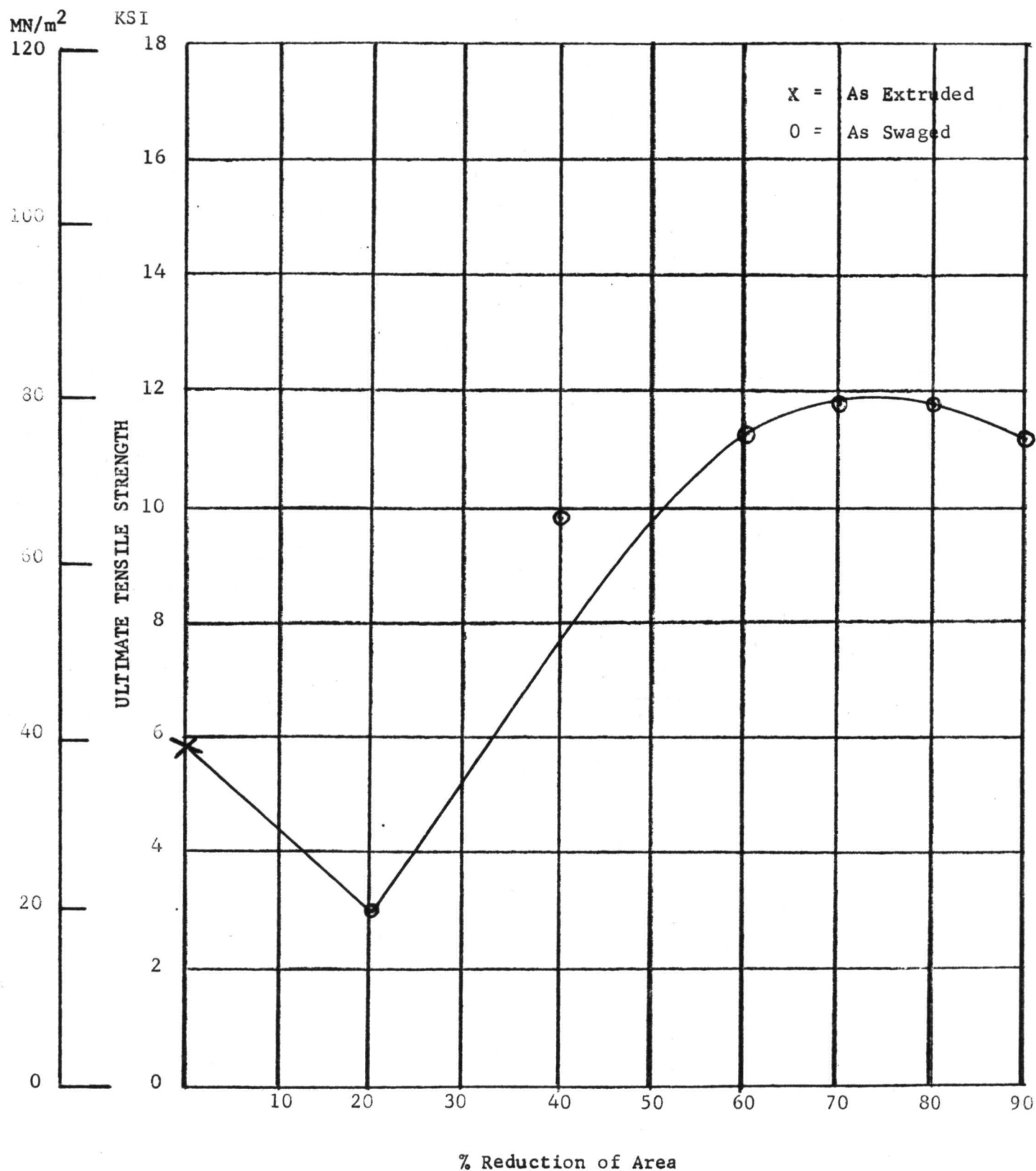
Photo. No. 9495  
J.R. No. 7111B  
Extrusion #2  
Front End

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C  
Heat Treat 4 Hrs. @ 1371°C

Mag. 10,000 X

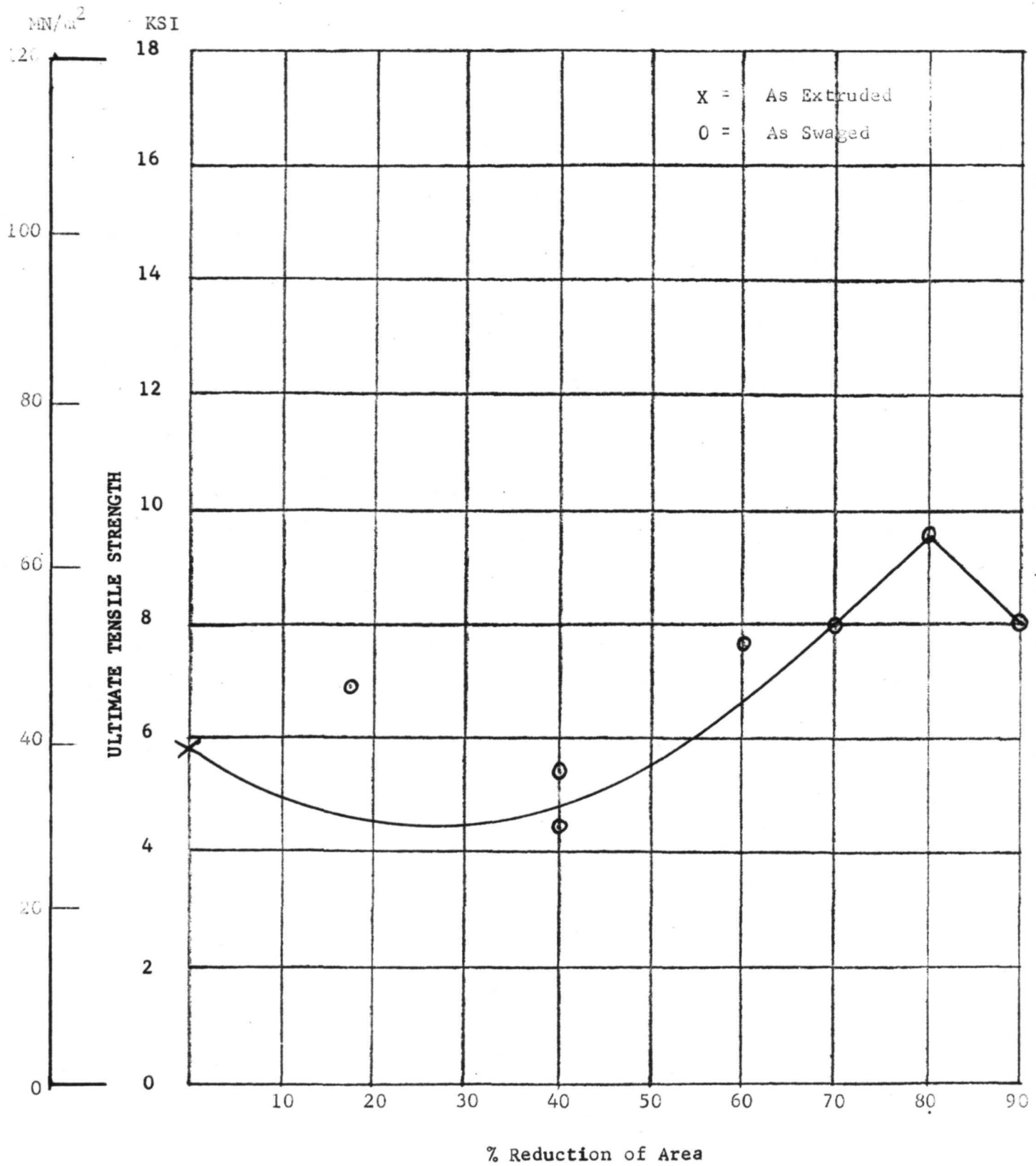
FIGURE 12

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69  
Extruded @ 10:1 @ 1094°C, Swaged @ 982°C





Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69  
Extruded @ 10:1 @ 1094°C, Swaged @ 1205°C



Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69  
Extruded @ 10:1 @ 1094°C, heat treated 2 hours @ 1316°C  
and swaged @ 982°C

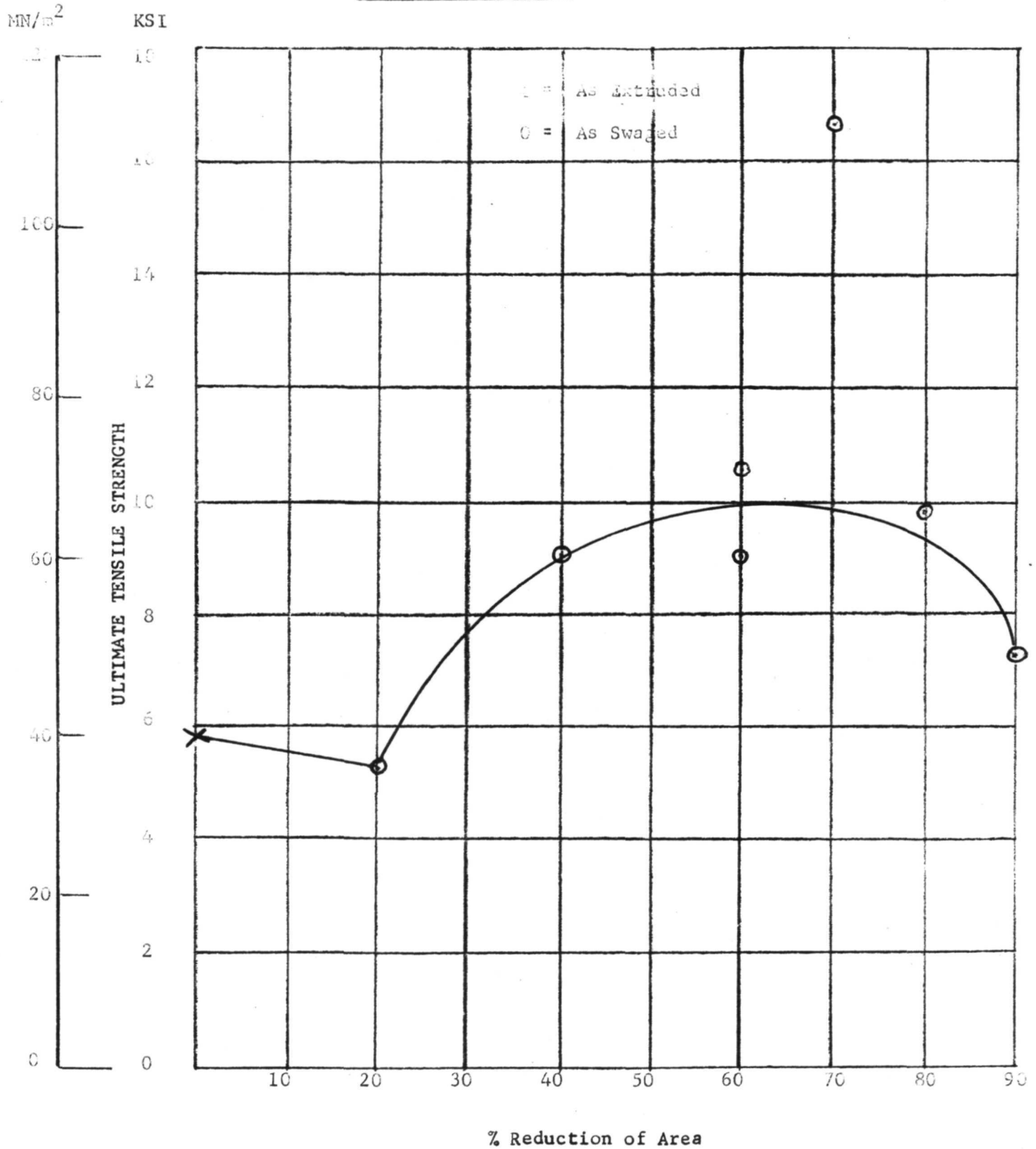
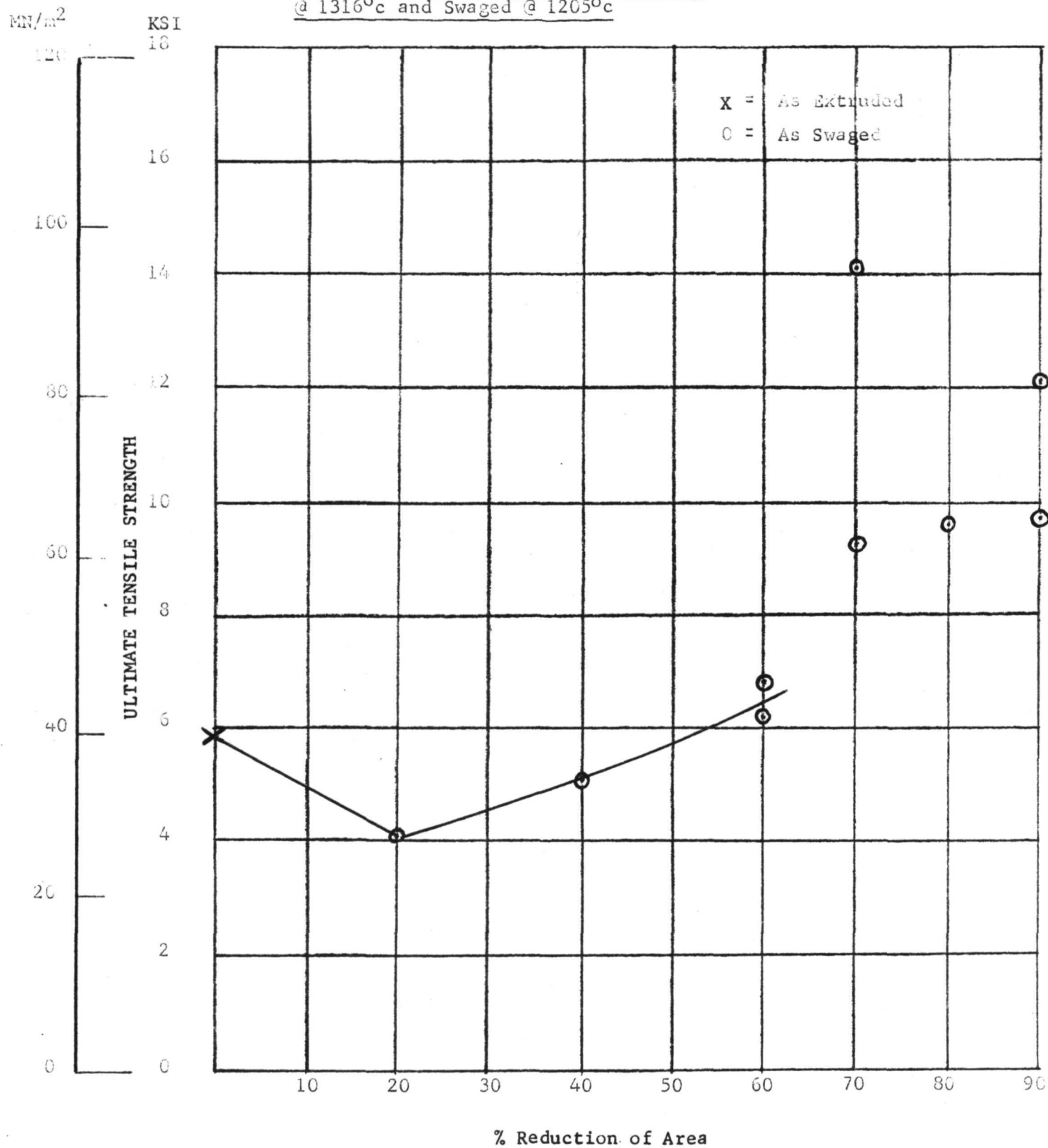


FIGURE 15

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-17A-69  
Extruded @ 10:1 @ 1094°C, Heat Treated 2 Hours  
@ 1316°C and Swaged @ 1205°C



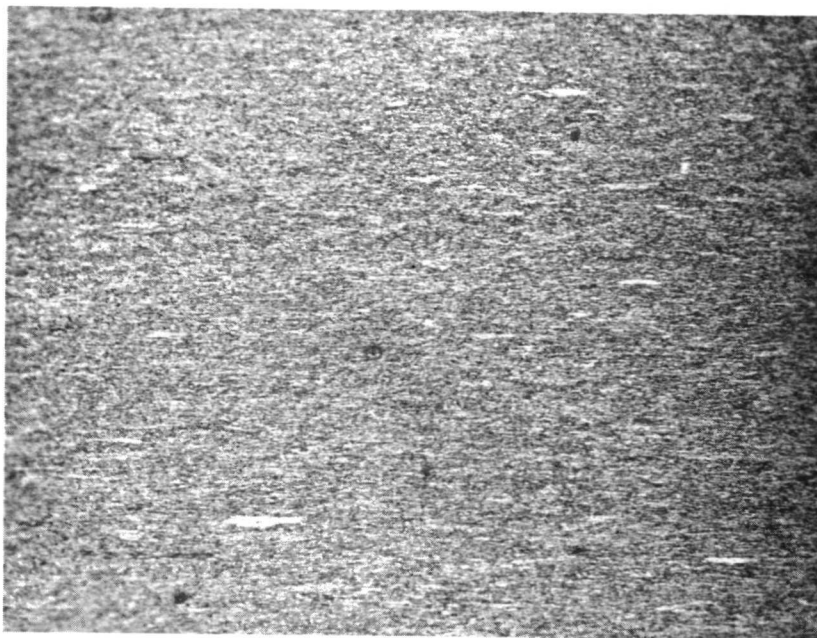


Figure 16A

Photo. No. P4723  
PP3-17A-69  
Extrusion #2  
(Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Density 100.4% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

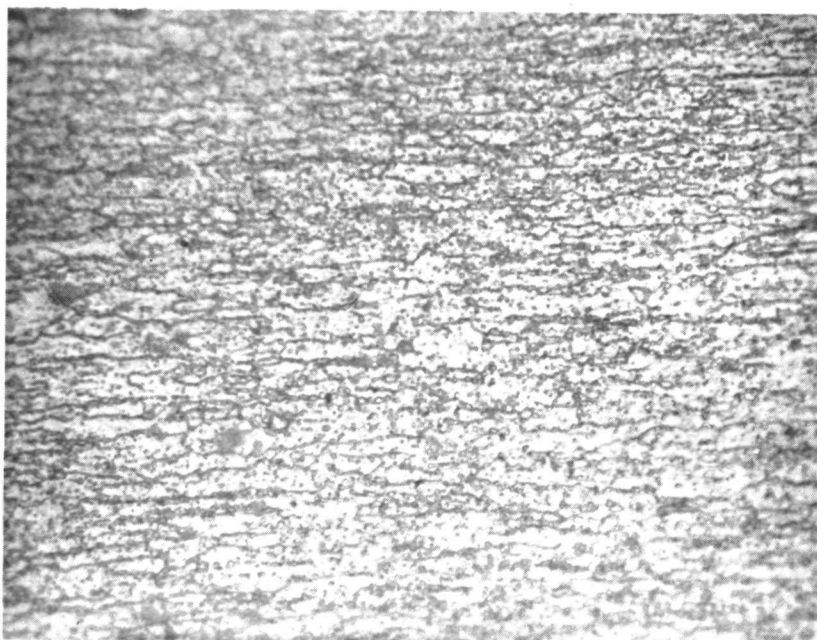


Figure 16B

Photo. No. P4724  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Density 100.4% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid

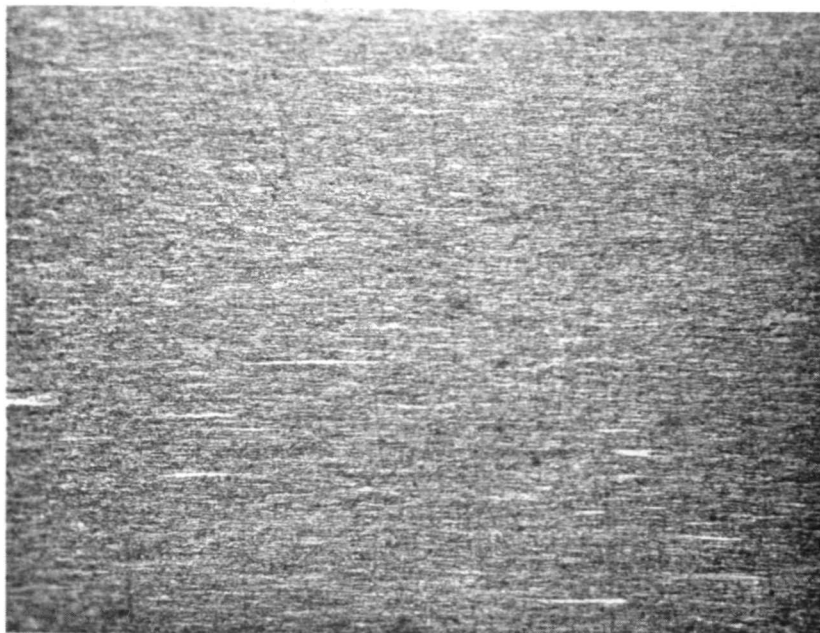


Photo. No. P4737  
PP3-17A-69  
Extrusion #2  
(Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Swaged 80% @ 1204°C  
Density 99.4% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

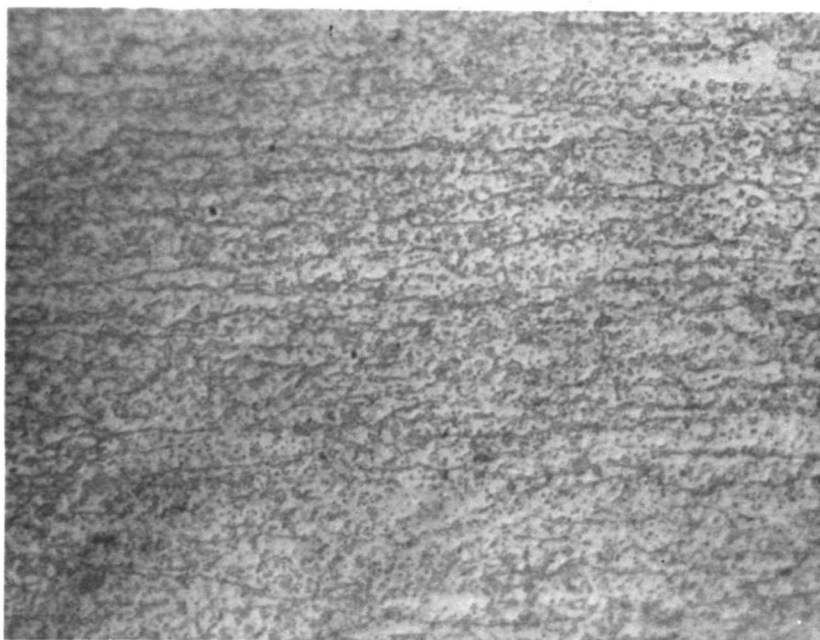


Figure 17B

Photo. No. P4738  
PP3-17A-69  
Extrusion #2  
(Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Swaged 80% @ 1204°C  
Density 99.4% of  
Theoretical Density

Mag. 1000X  
Etchant: Electrolytic  
Perchloric Acid



Figure 18A

Photo. No. P4747  
PP3-17A-69  
Extrusion #2  
(Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 70% @ 982°C  
Density 99.3% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

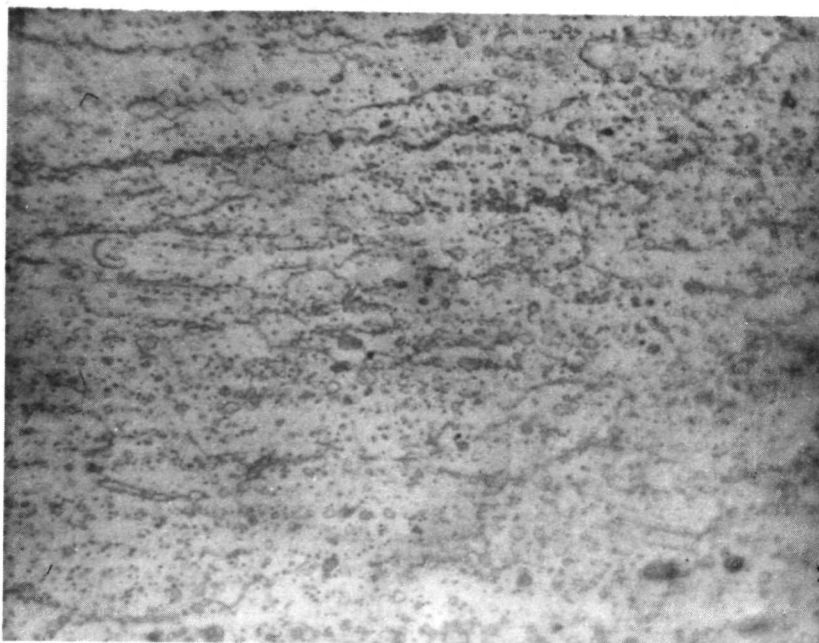


Figure 18B

Photo. No. P4748  
PP3-17A-69  
Extrusion #2  
(Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 70% @ 982°C  
Density 99.3% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid





Figure 19A

Photo. No. P4761  
PP3-17A-69  
Extrusion #2  
(Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 80% @ 1204°C  
Density 99.2% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

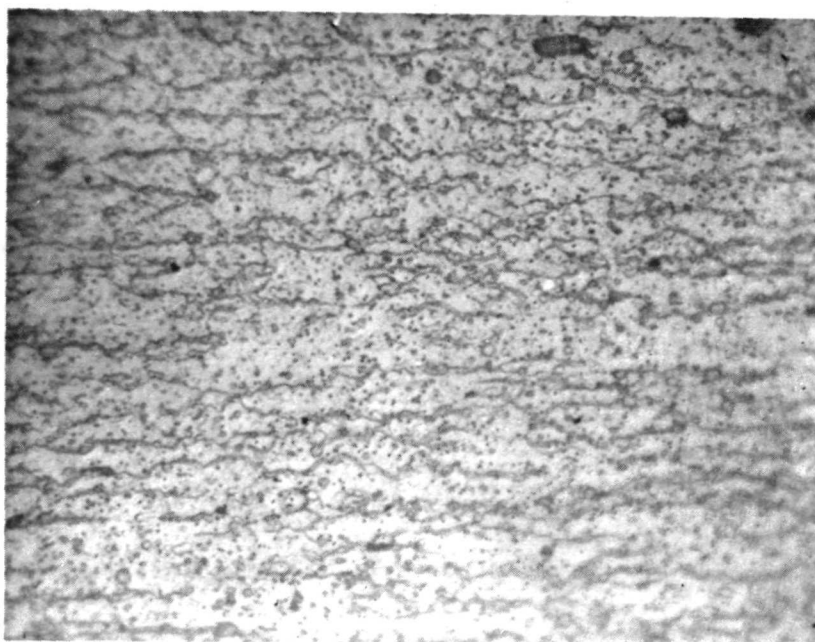


Figure 19B

Photo. No. P4762  
PP3-17A-69  
Extrusion #2  
(Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 80% @ 1204°C  
Density 99.2% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 20A

Photo. No. P4767  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Swaged 60% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.8% of Theoretical  
Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

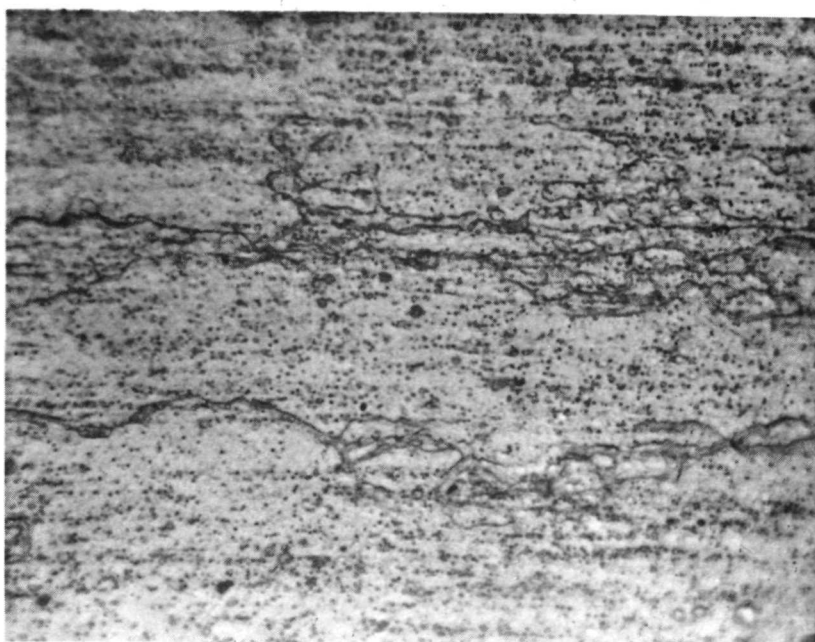


Figure 20B

Photo. No. P4768  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Swaged 60% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.8% of Theoretical  
Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



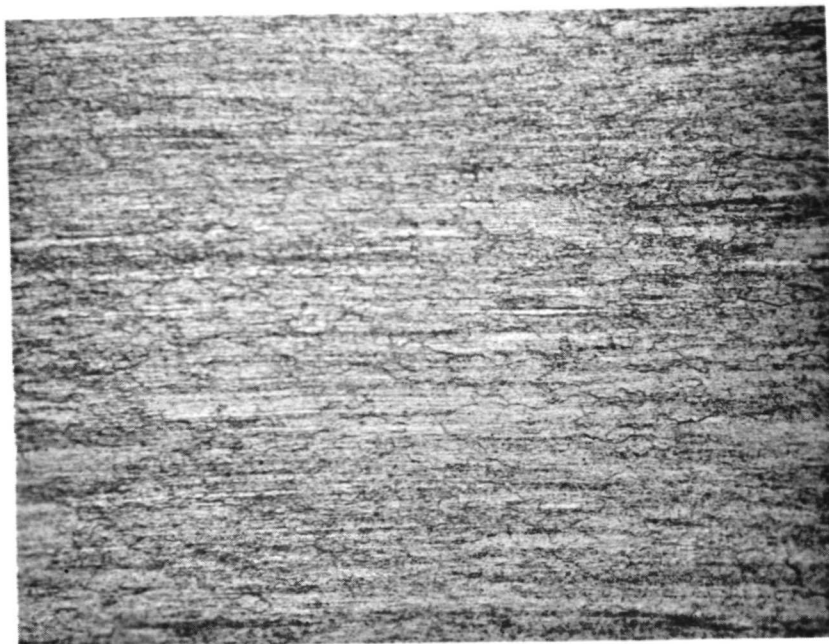


Figure 21A

Photo. No. P4773  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Heat Treat 2 Hours @ 1371°C

Mag. 100 X  
Etchant: Electrolytic  
Perchloric acid



Figure 21B

Photo. No. P4774  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Heat Treat 2 Hours @ 1371°C

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 22A

Photo. No. P4791  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 90% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.42% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

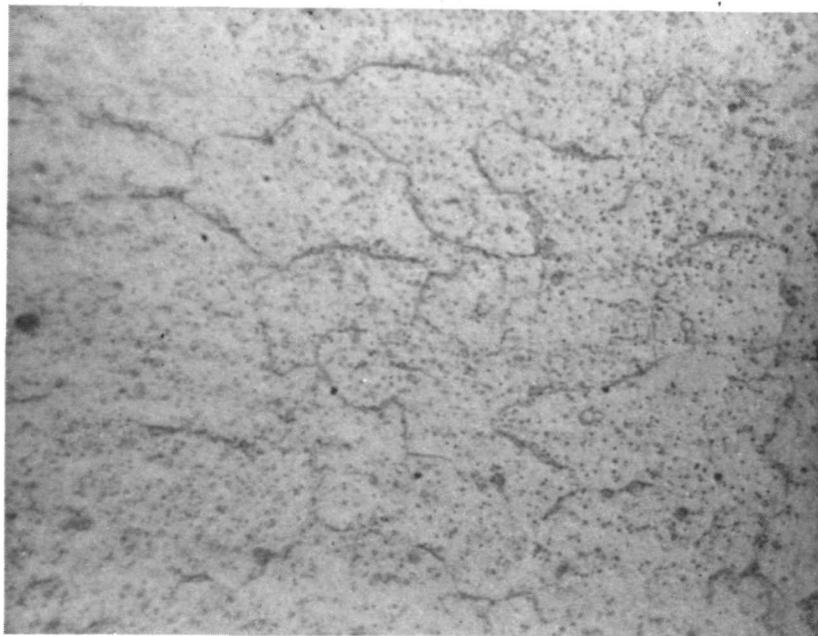


Figure 22B

Photo. No. P4792  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 90% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.42% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 23A

Photo. No. P4775  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 80% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.72% of Theoretical  
Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

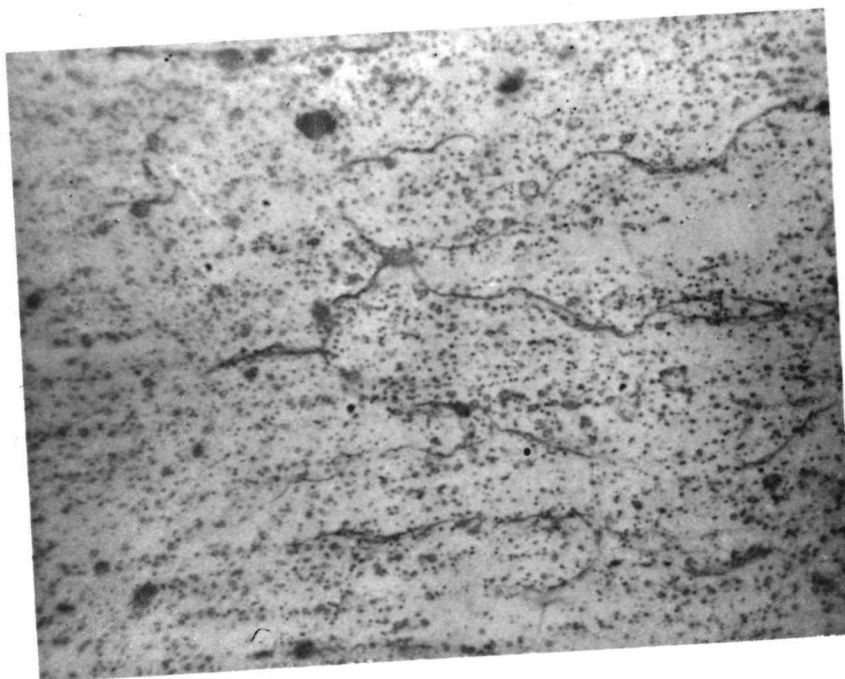


Figure 23B

Photo. No. P4776  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 80% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.72% of Theoretical  
Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 24A

Photo. No. P4803  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 60% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.78%  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

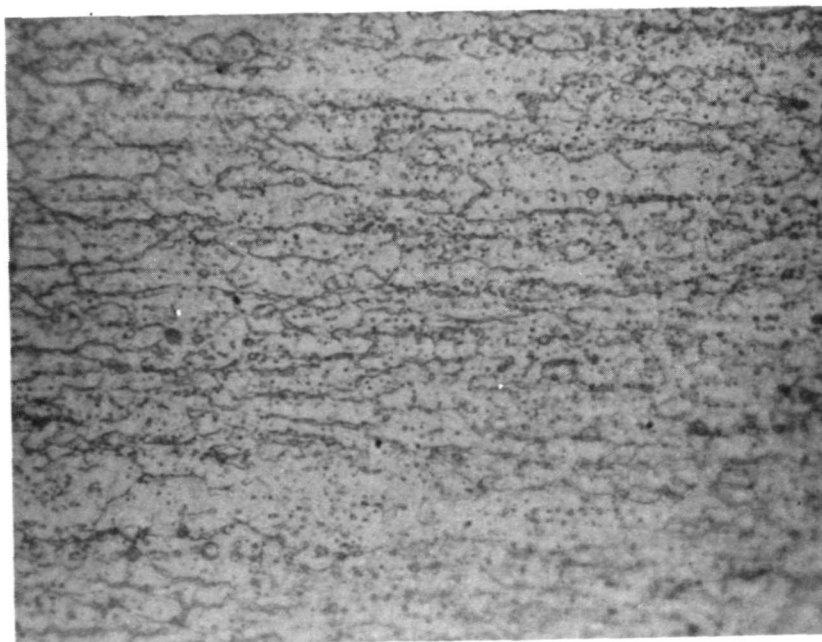


Figure 24B

Photo. No. P4804  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 60% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.78%  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 25A

Photo. No. P4777  
 PP3-17A-69  
 Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
 Extruded 10:1 @ 1094°C  
 Swaged 70% @ 1204°C  
 Heat Treat 2 Hours @ 1371°C  
 Density 99.72% of  
 Theoretical Density

Mag. 100X  
 Etchant: Electrolytic  
 Perchloric Acid

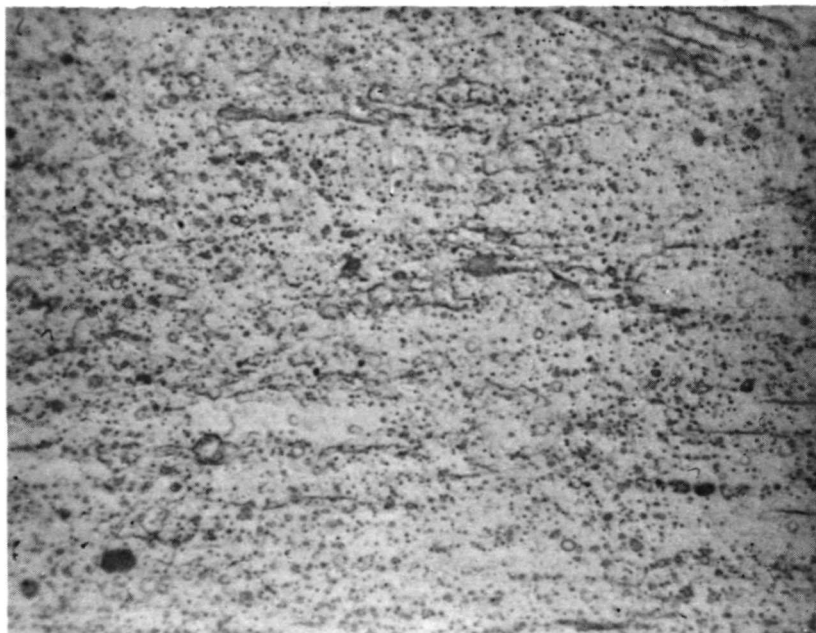


Figure 25B

Photo. No. P4778  
 PP3-17A-69  
 Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
 Extruded 10:1 @ 1094°C  
 Swaged 70% @ 1204°C  
 Heat Treat 2 Hours @ 1371°C  
 Density 99.72% of  
 Theoretical Density

Mag. 1000 X  
 Etchant: Electrolytic  
 Perchloric Acid



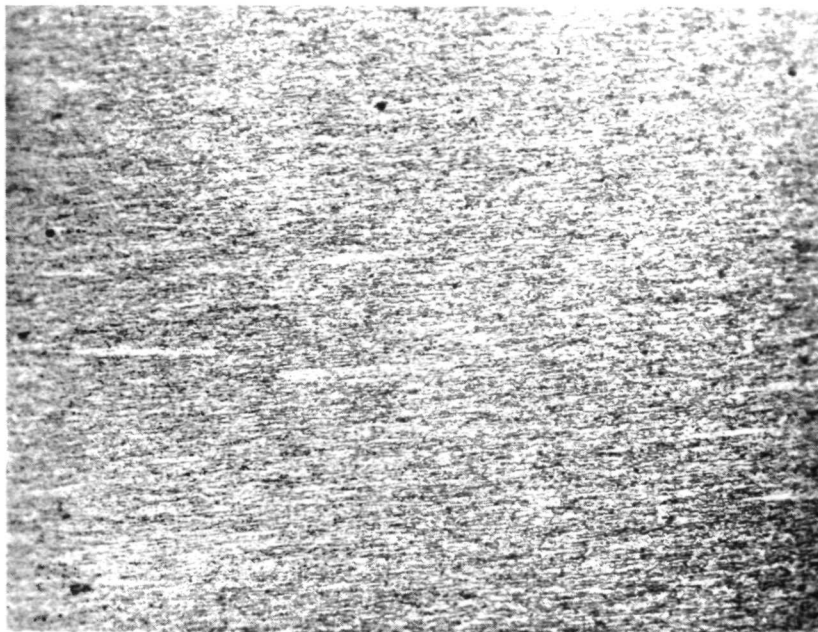


Figure 26A

Photo. No. P4799  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 1204°C  
Heat Treat 4 Hours @ 1371°C  
Density 99.71% of  
Theoretical Density

Mag. 160 X  
Etchant: Electrolytic  
Perchloric Acid

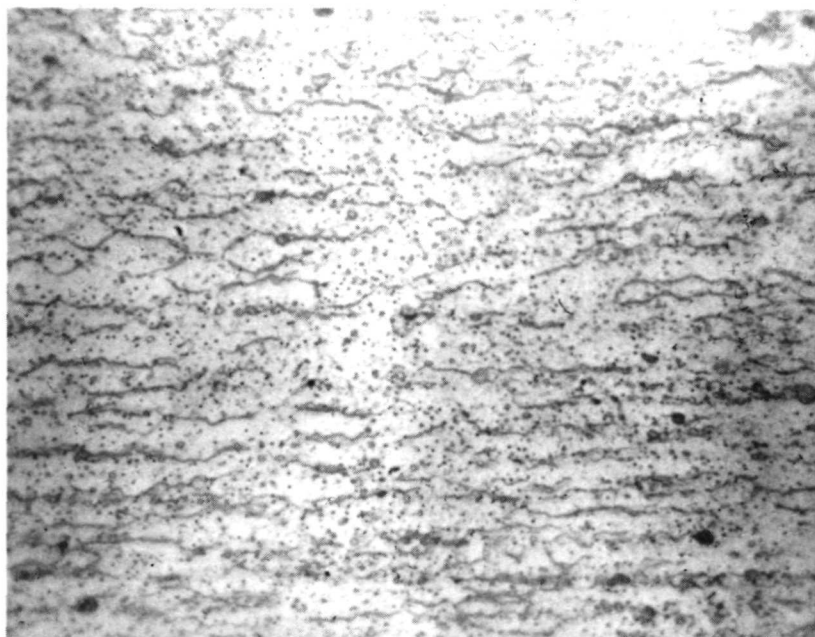


Figure 26B

Photo. No. P4800  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 1204°C  
Heat Treat 4 Hours @ 1371°C  
Density 99.71% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid

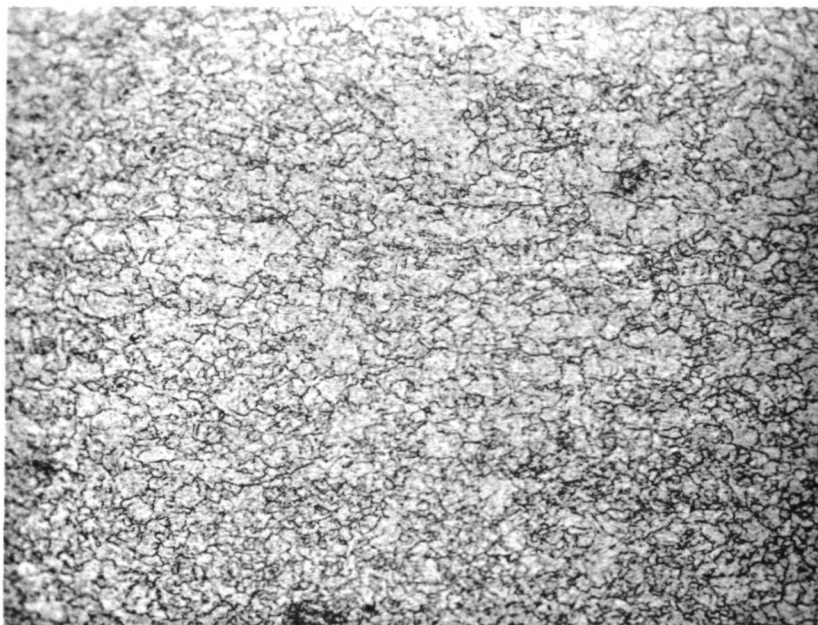


Figure 27A

Photo. No. P4769  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Swaged 90% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.43% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

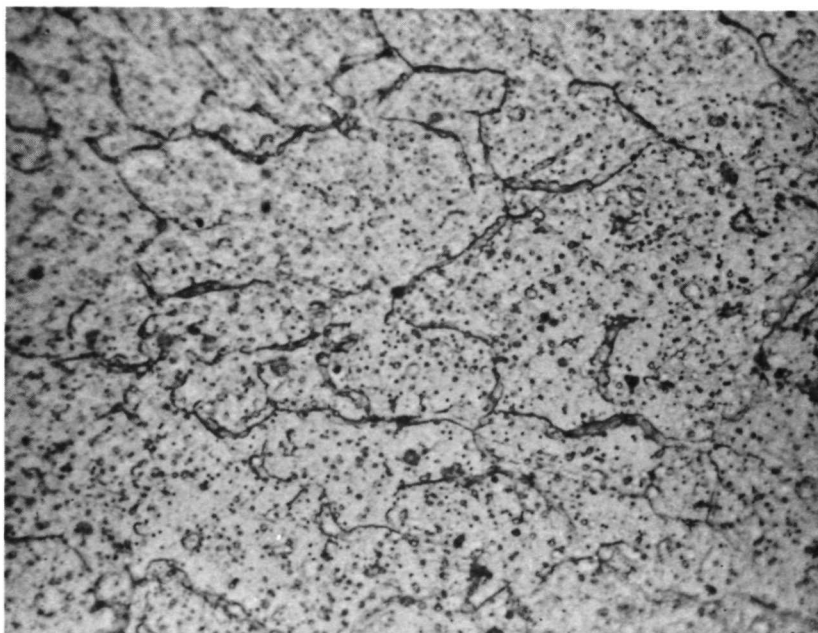


Figure 27B

Photo. No. P4770  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 @ 1094°C  
Swaged 90% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.43% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 28A

Photo. No. P4779  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 60% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.54% of

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 28B

Photo. No. P4780  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 60% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.54% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



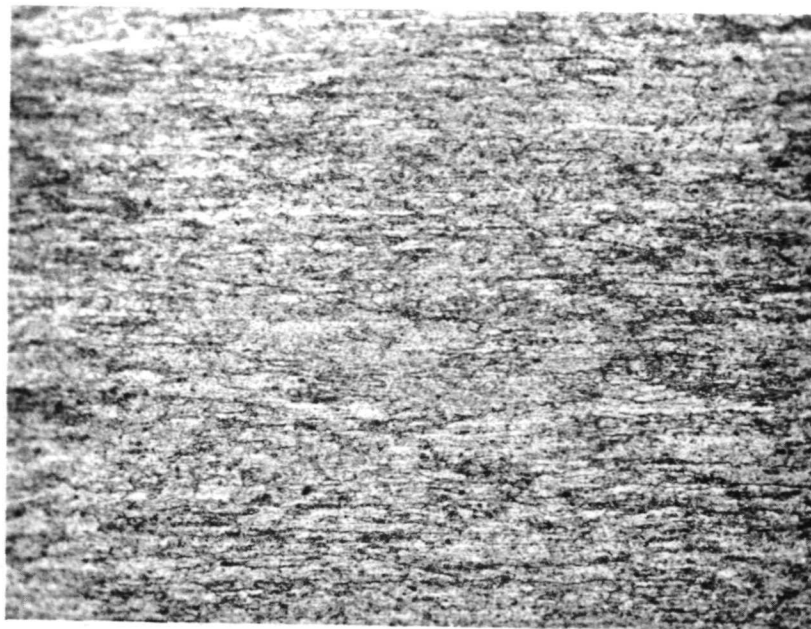


Figure 29A

Photo. No. P4771  
PP3-17A-69  
Extrusion #1 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Heat Treat 2 hours @ 1316°C  
Swaged 70% @ 982°C  
Heat Treat 2 hours @ 1371°C  
Density 99.45% of  
Theoretical Density

Mag. 100X  
Etchant: Electrolytic  
Perchloric Acid

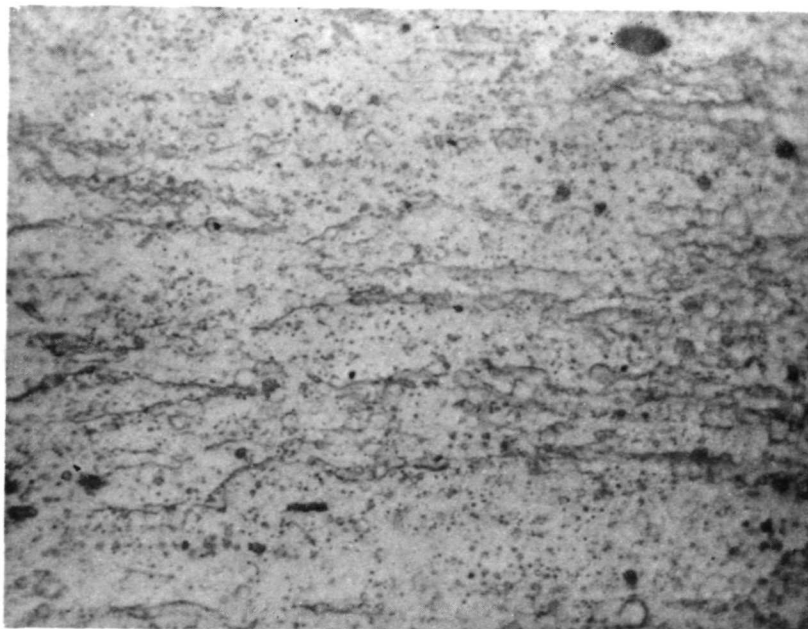


Figure 29B

Photo. No. P4772  
PP3-17A-69  
Extrusion #1 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Heat Treat 2 hours @ 1316°C  
Swaged 70% @ 982°C  
Heat Treat 2 hours @ 1371°C  
Density 99.45% of  
Theoretical Density

Mag. 100X  
Etchant: Electrolytic  
Perchloric Acid

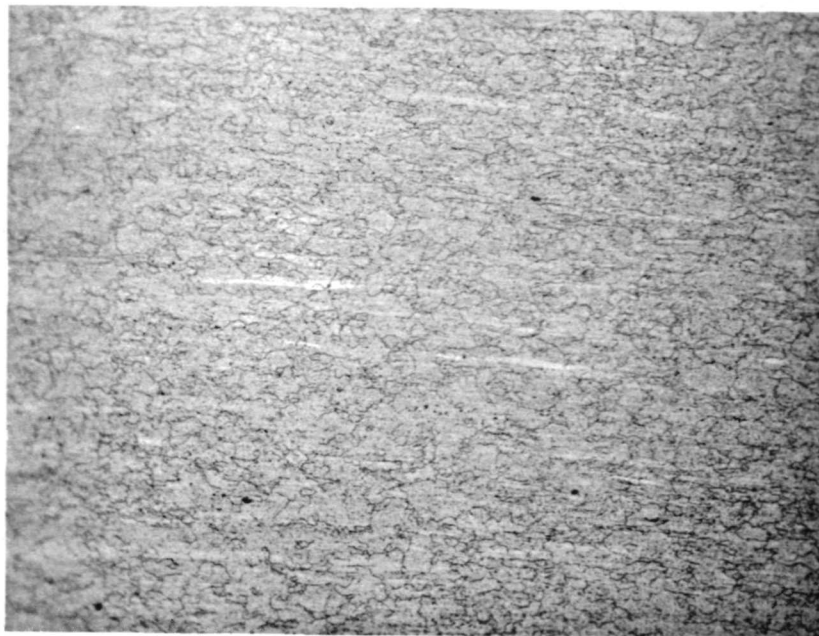


Figure 30A

Photo. No. P4795  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Heat Treat 2 hours @ 1316°C  
Swaged 80% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.55% of  
Theoretical Density

Mag. 100X  
Etchant: Electrolytic  
Perchloric Acid

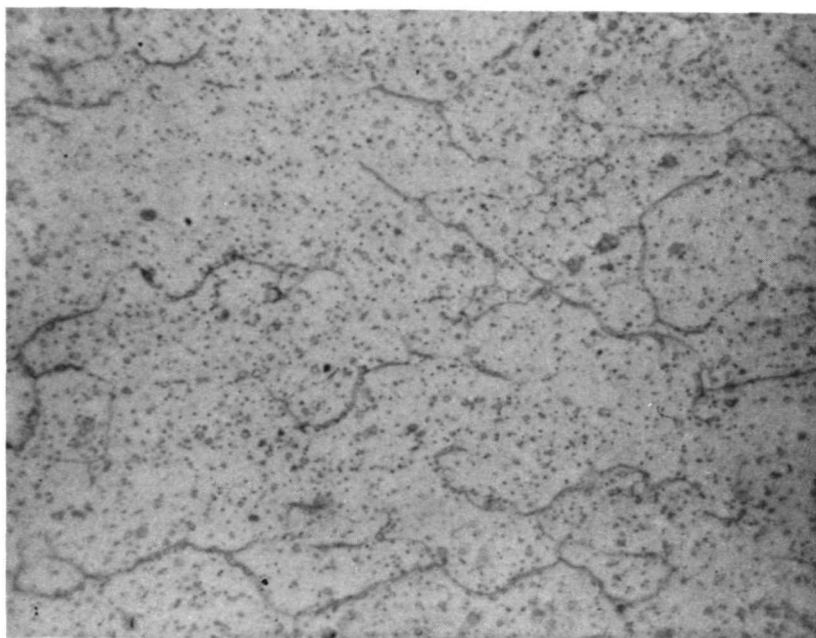


Figure 30B

Photo. No. P4796  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Heat Treat 2 hours @ 1316°C  
Swaged 80% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.55% of  
Theoretical Density

Mag. 1000X  
Etchant: Electrolytic  
Perchloric Acid

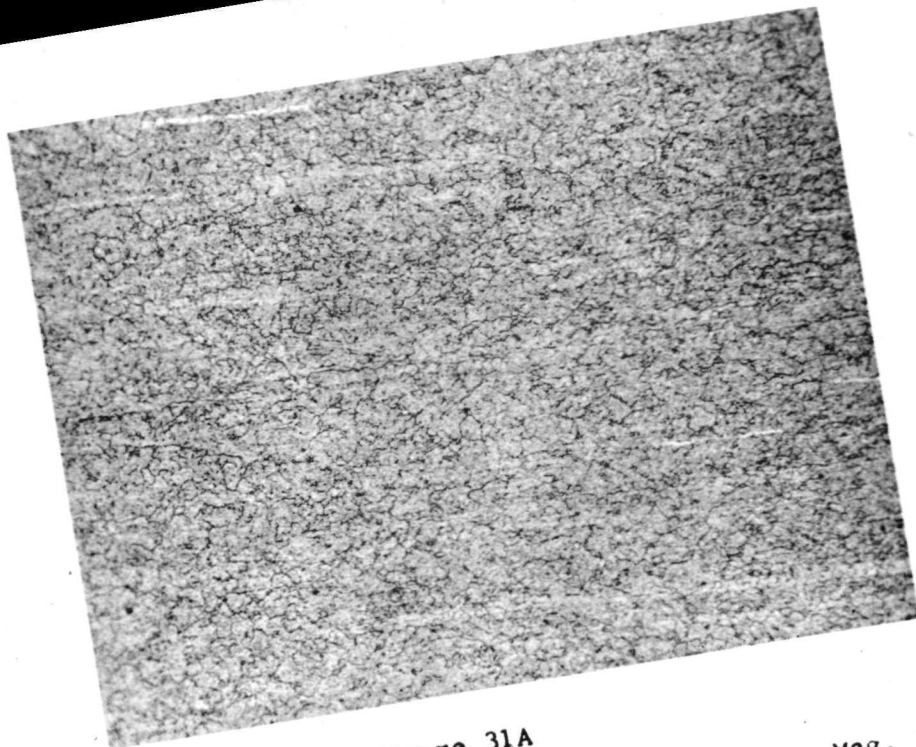


Figure 31A

Photo. No. P4809  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6 ThO<sub>2</sub>  
Extruded @ 10:1 Ratio @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 90% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 100.24%  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

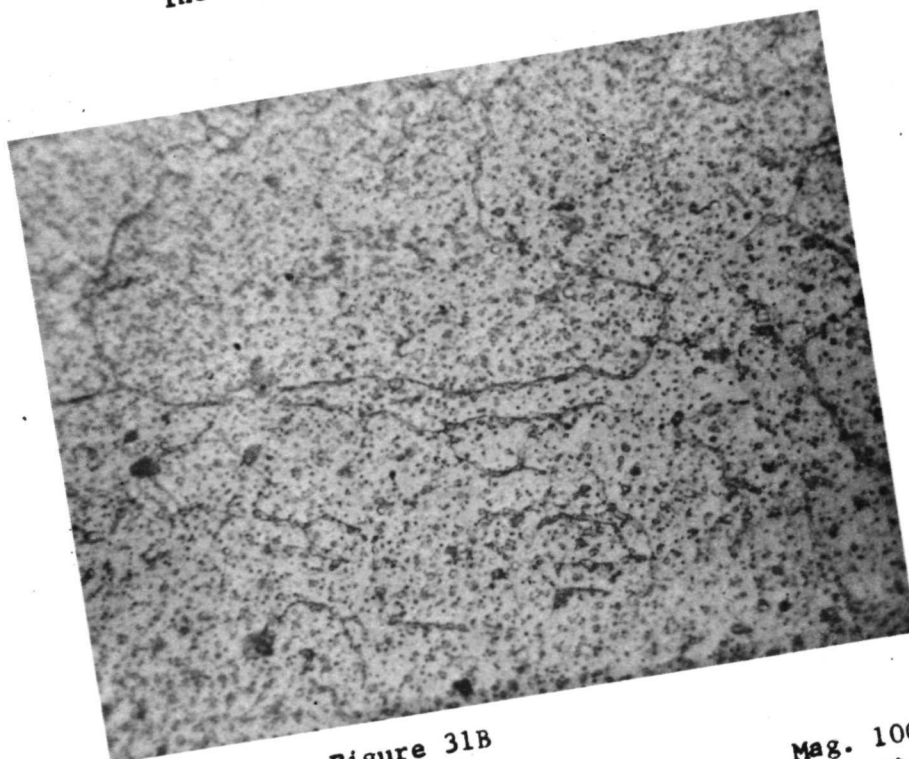


Figure 31B

Photo. No. P4810  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded @ 10:1 Ratio 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 90% @ 982°C  
Heat Treat 2 Hours @ 1371°C  
Density 100.24%  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid

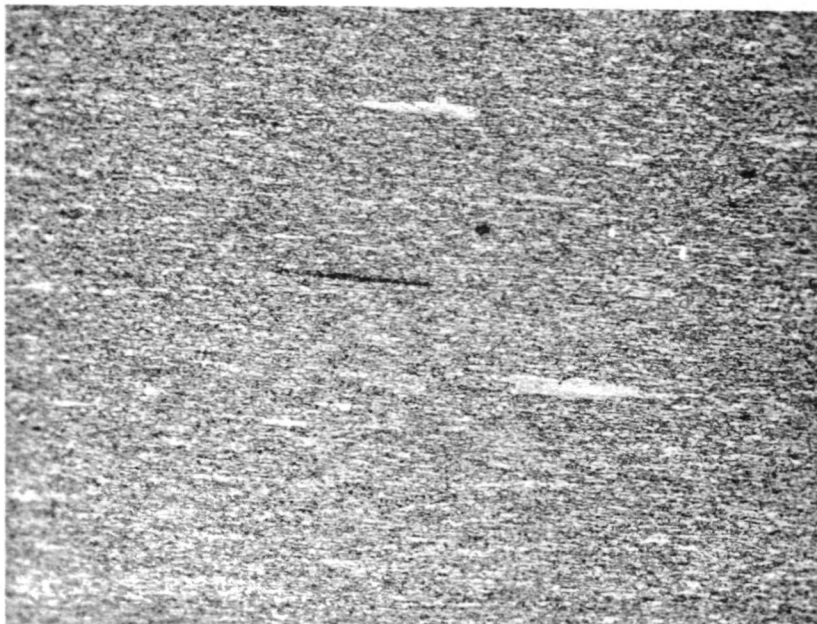


Figure 32A

Photo. No. P4807  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 Ratio @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 60% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.75% of

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

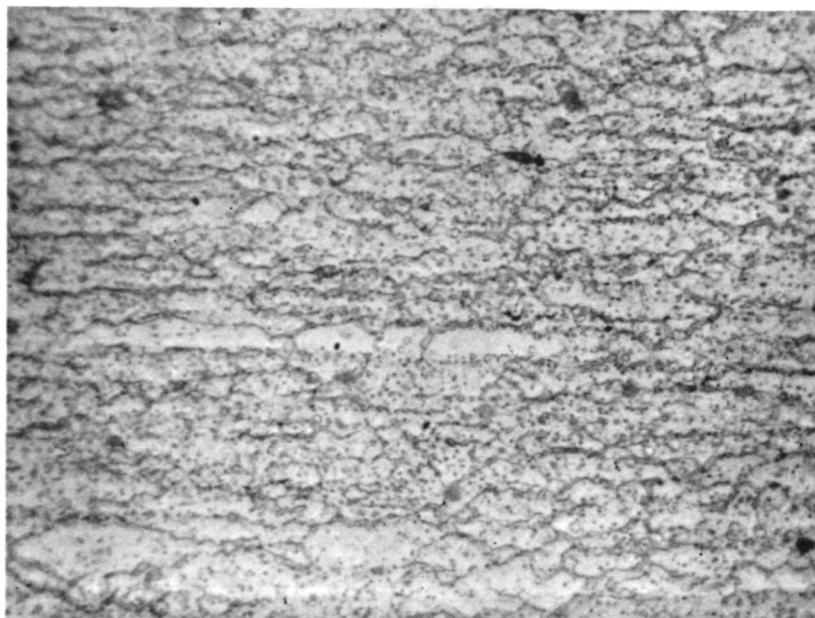


Figure 32B

Photo. No. P4808  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 Ratio @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 60% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.75% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 33A

Photo. No. P4781  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 70% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.39% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

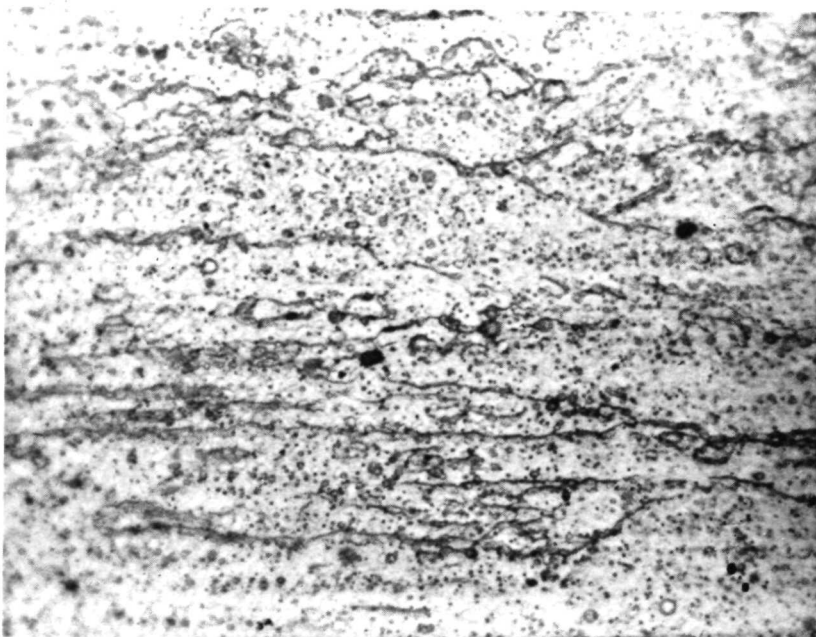


Figure 33B

Photo No. P4782  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 70% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.39% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid





Figure 34A

Photo. No. P4783  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1204°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 80% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.46% of

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 34B

Photo. No. P4784  
PP3-17A-69  
Extrusion #2 (Back)

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1204°C  
Heat Treat 2 Hours @ 1316°C  
Swaged 80% @ 1204°C  
Heat Treat 2 Hours @ 1371°C  
Density 99.46% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid

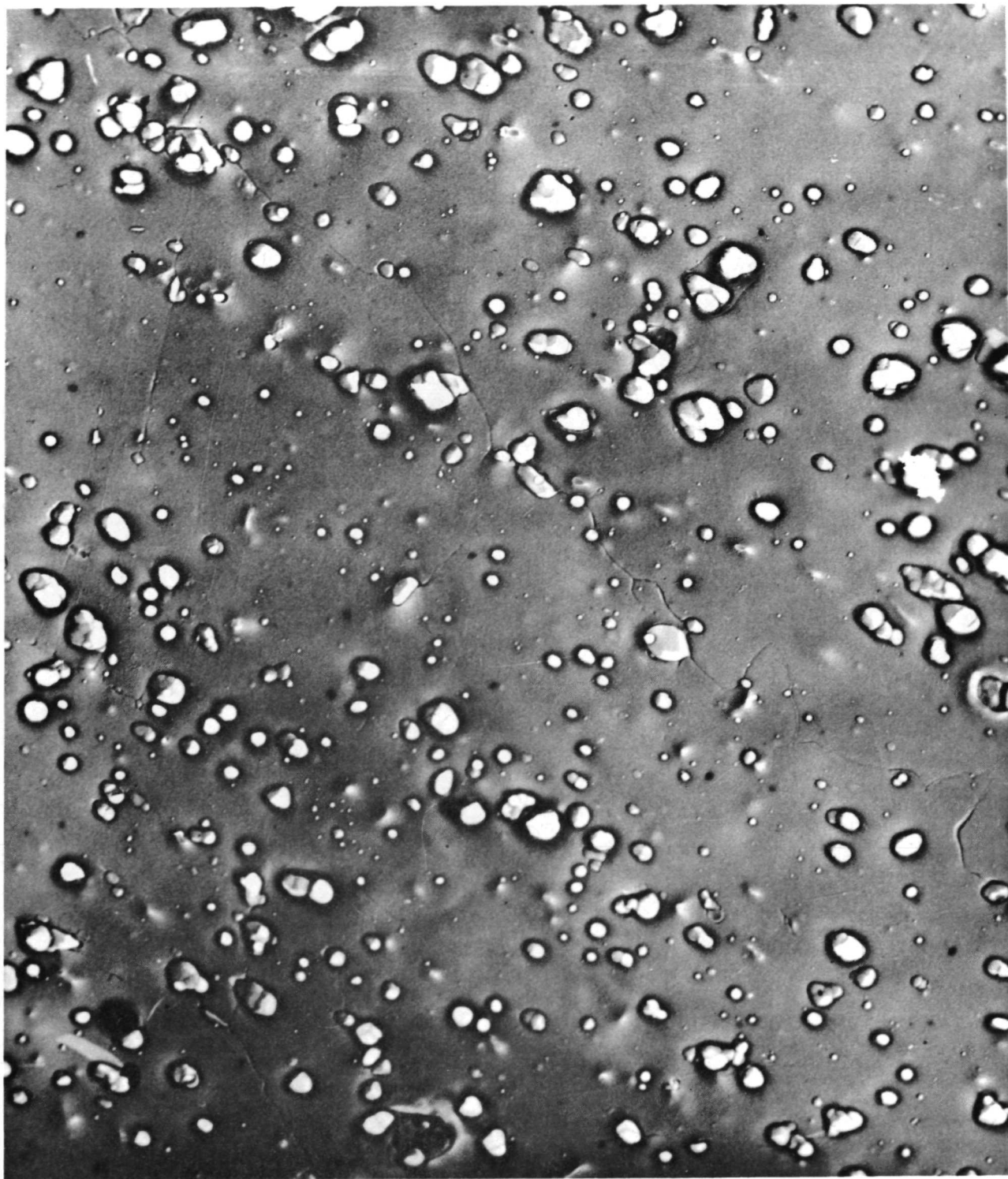


Figure 35

Photo. No. 9613  
J.R. No. 7224  
Extrusion #1

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C  
Swaged to 60% R.A. @ 982°C  
Annealed at 1204°C after each  
swaging step from 0% R.A.

Mag. 10,000 X

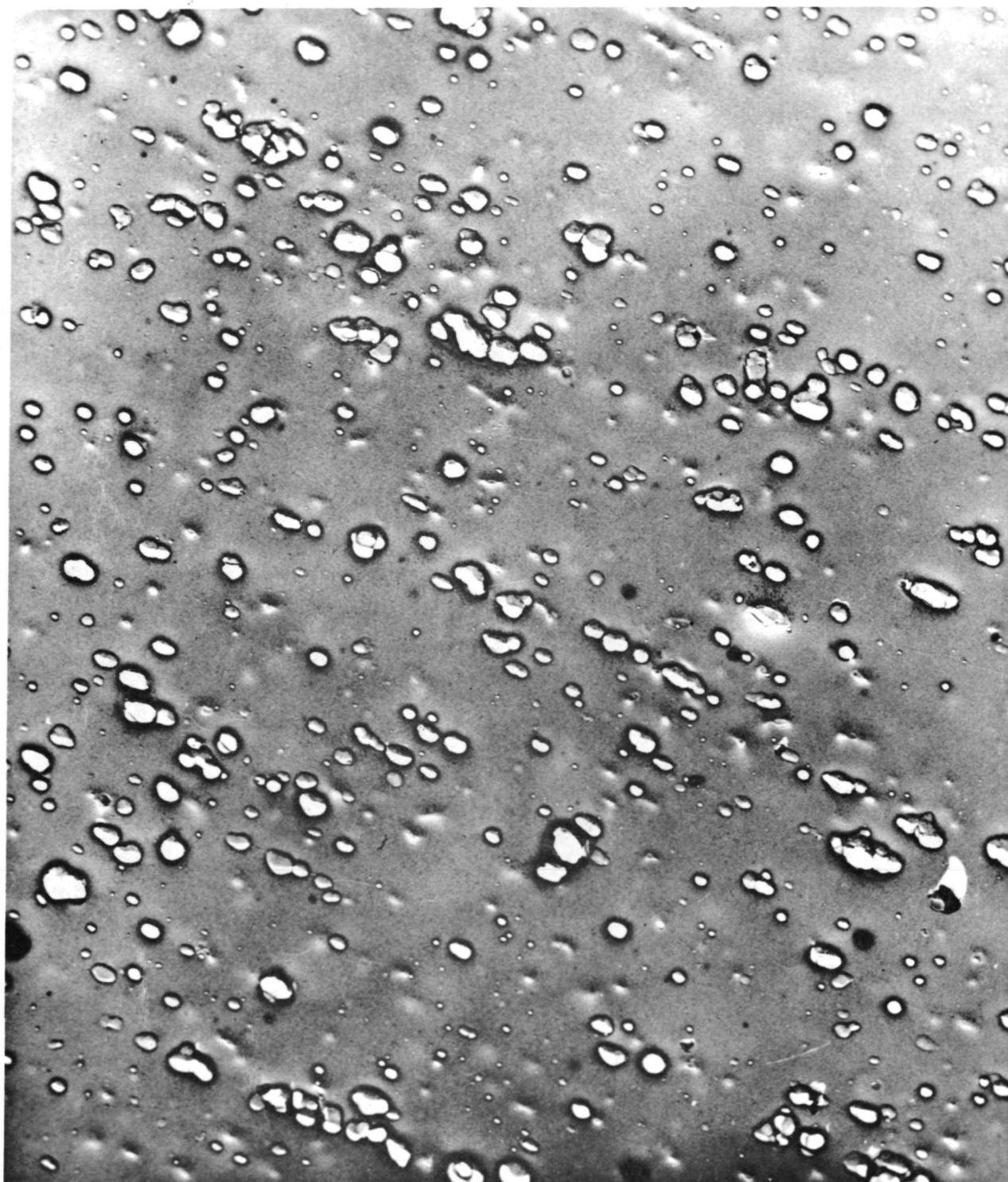


Figure 36

Photo. No. 9614  
J.R. No. 7225  
Extrusion #2

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3      Mag. 10,000 X  
Extruded 10:1 @ 1094°C  
Swaged to 60% R.A. @ 982°C  
Annealed at 1204°C after each swaging  
step from 40% R.A. and heat treat 2  
hours @ 1371°C.



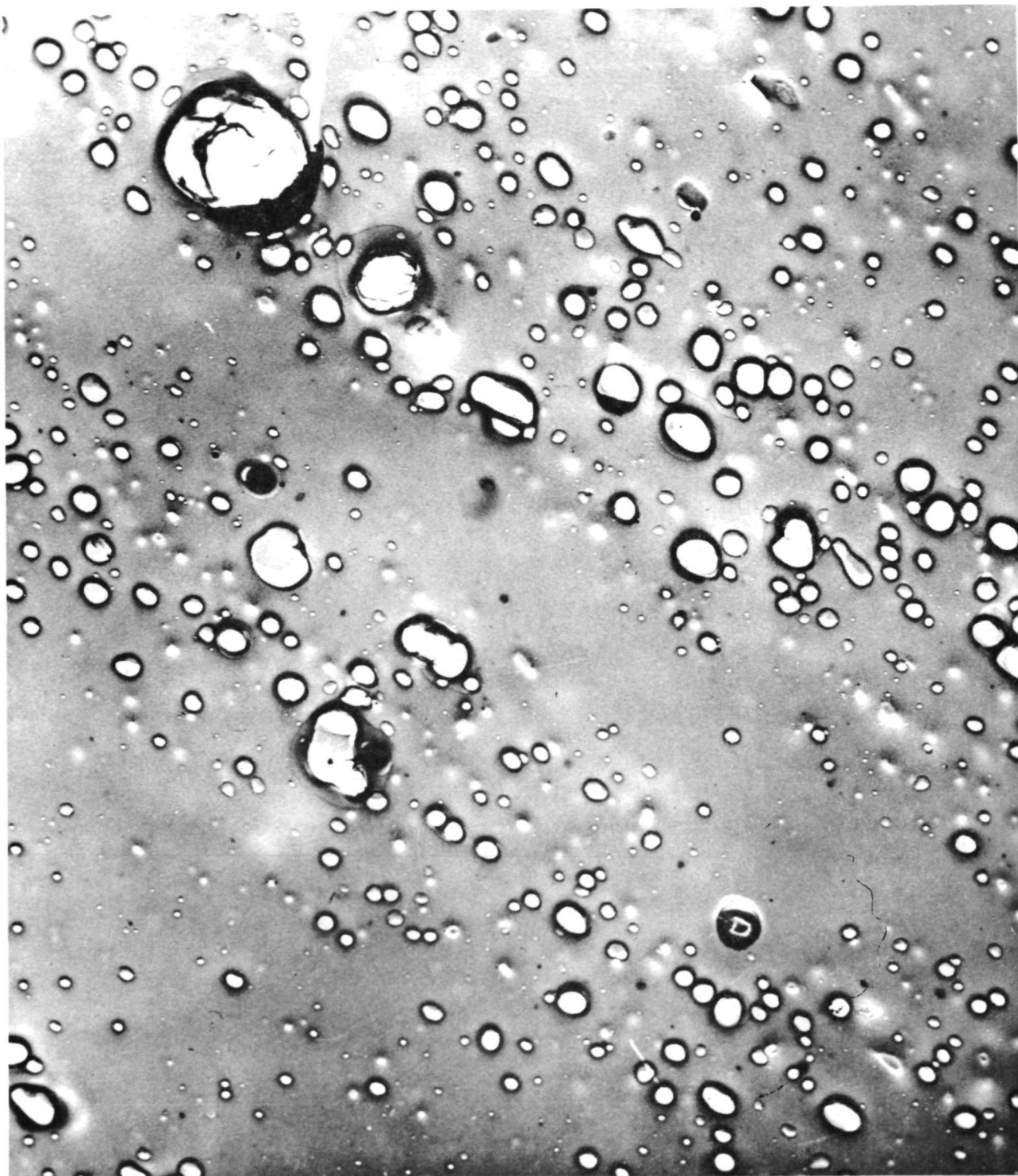


Figure 37

Photo. No. 9615  
J.R. No. 7226  
Extrusion #2

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C  
Swaged to 70% R.A. @ 982°C  
Annealed at 1204°C after each  
swaging step from 40% R.A.

Mag. 10,000 X

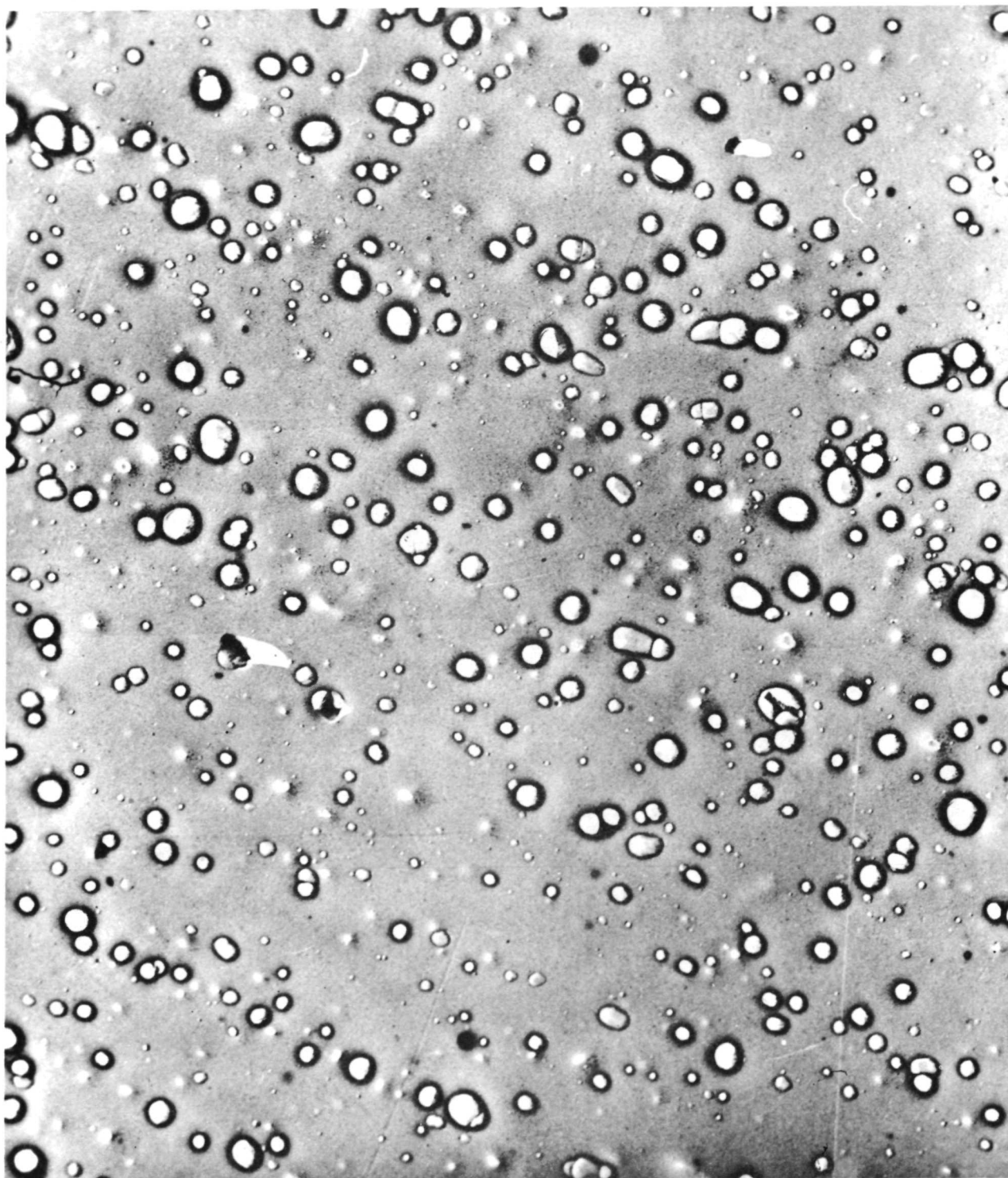


Figure 38

Photo. No. 9616  
J.R. No. 7227  
Extrusion #2

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C  
Swaged to 70% R.A. @ 982°C  
Annealed at 1204°C after each  
swaging step from 40% R.A. and heat  
treat 2 hours @ 1371°C

Mag. 10,000 X

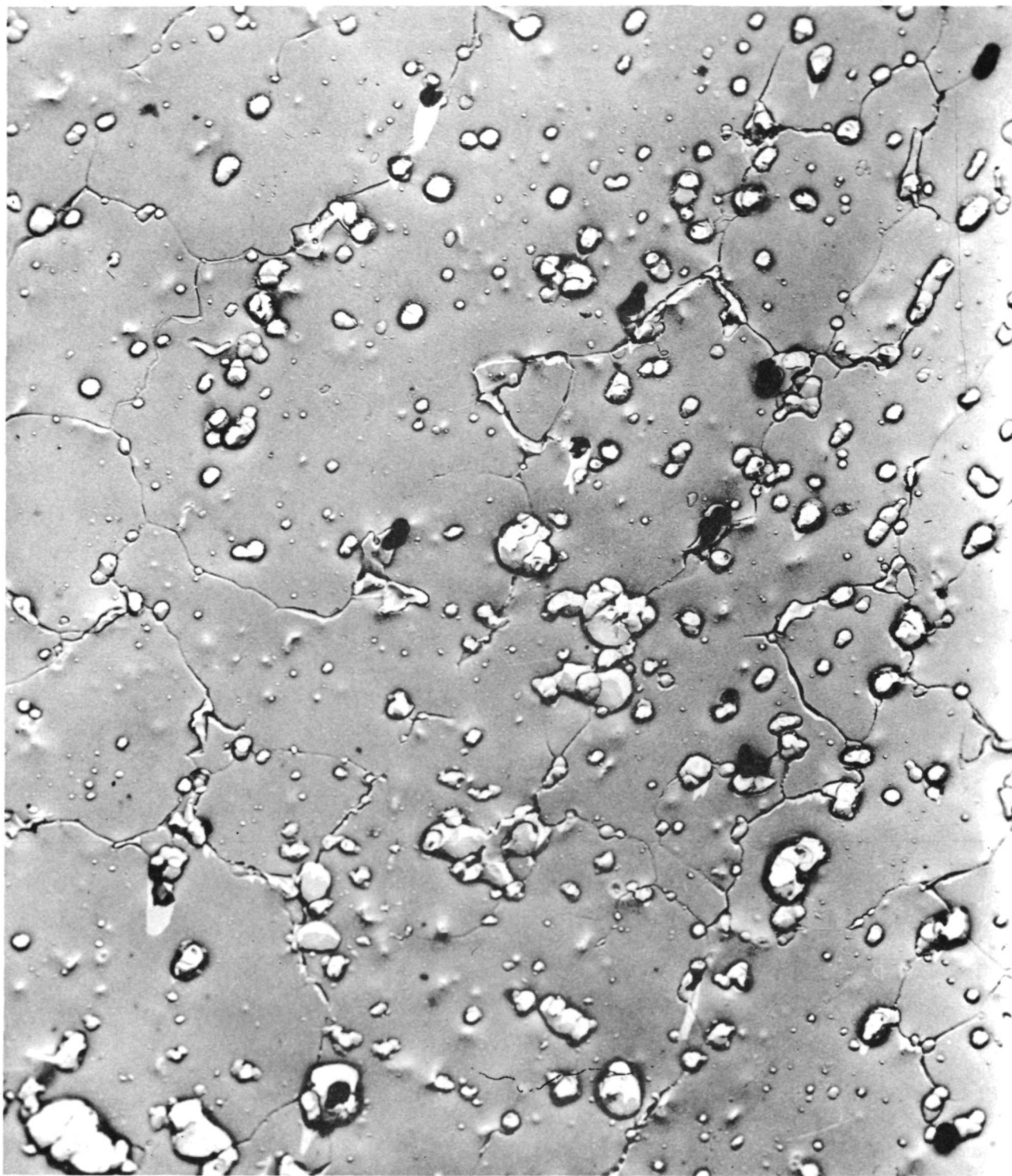


Figure 39

Photo. No. 9617  
J.R. No. 7228  
Extrusion #2

Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3  
Extruded 10:1 @ 1094°C  
Swaged to 60% R.A. @ 1204°C

Mag. 10,000 X



Figure 40A

Photo. No. P4819  
PP3-13A-76  
Extrusion #6  
Route 3

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Density 99.50% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 40B

Photo. No. 4820  
PP3-13A-76  
Extrusion #6  
Route 3

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Density 99.50% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



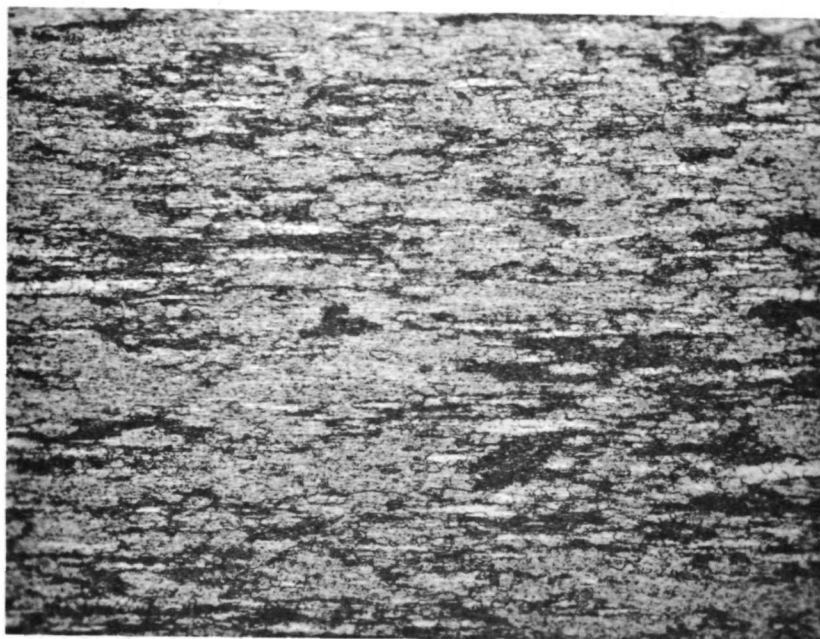


Figure 41A

Photo. No. P4899  
PP3-13A-76  
Extrusion #6  
Route 3

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Heat Treat 10-1/2 hours @ 1204°C  
Density 99.47% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 41B

Photo. No. 4900  
PP3-13A-76  
Extrusion #6  
Route 3

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Heat Treat 10-1/2 hours @ 1204°C  
Density 99.47% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 42A

Photo. No. P4905  
PP3-13A-76  
Extrusion #6  
Route 3

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Heat Treat 10-1/2 hours @ 1204°C  
Density 99.4% of  
Theoretical Density

Mag. 100 X  
Etchant: Electrolytic  
Perchloric Acid

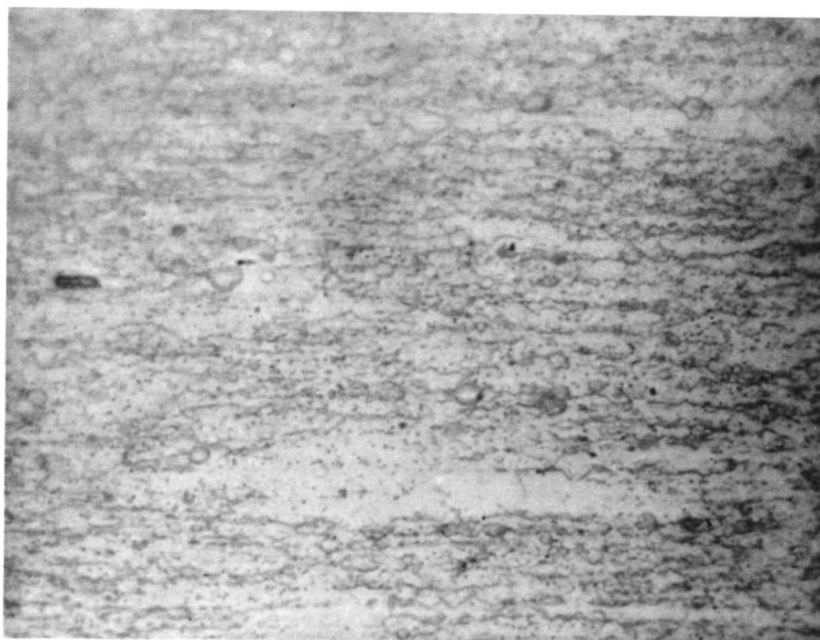


Figure 42B

Photo. No. P4906  
PP3-13A-76  
Extrusion #6  
Route 3

Co-18Cr-20Ni-4.6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged 70% @ 982°C  
Heat Treat 10-1/2 hours @ 1204°C  
Density 99.4% of  
Theoretical Density

Mag. 1000 X  
Etchant: Electrolytic  
Perchloric Acid



Figure 43

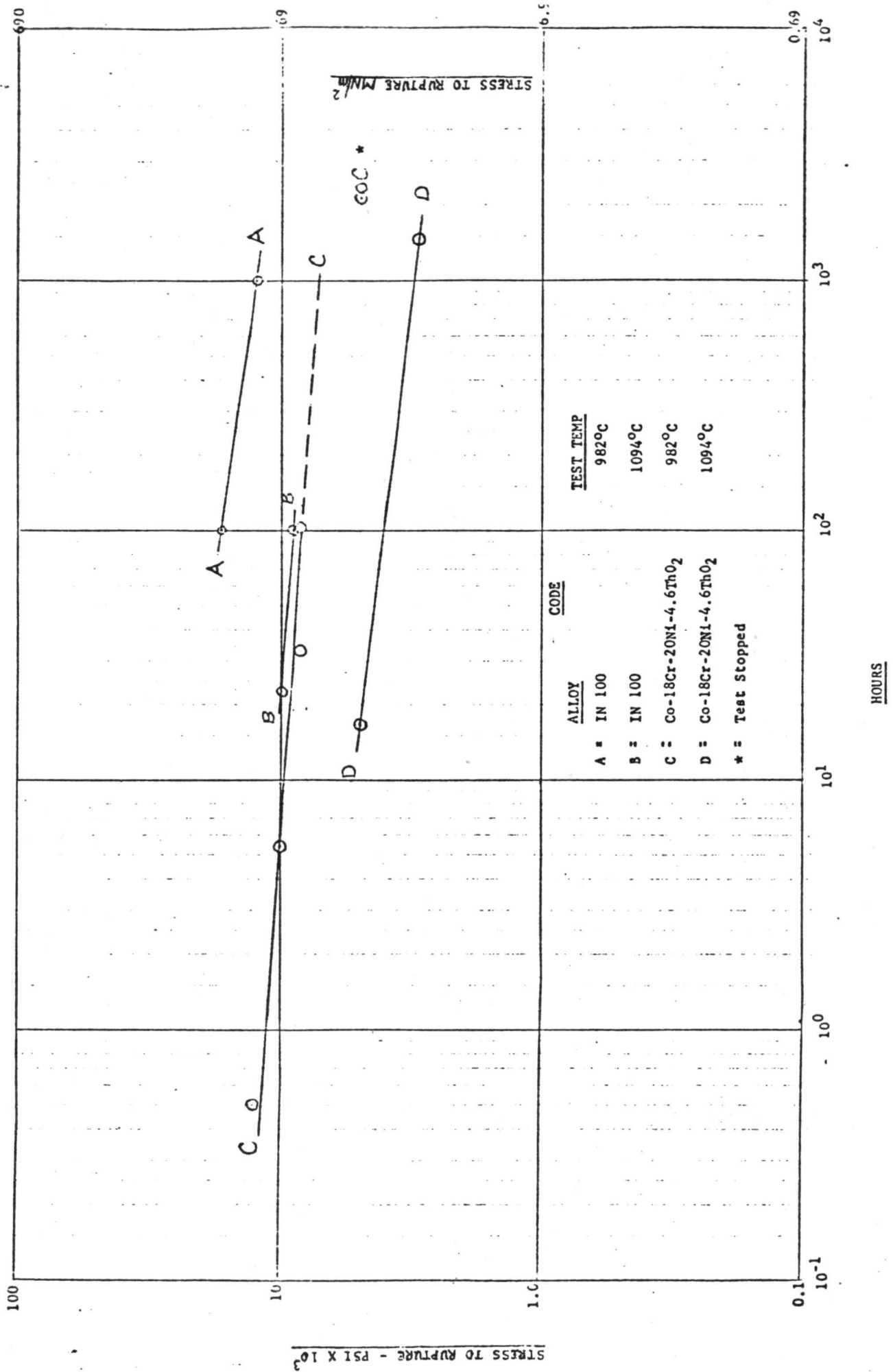
Photo. No. 9734  
J.R. No. 7252  
PP3-13A-76  
Extrusion #6

Co-18Cr-20Ni-4 6ThO<sub>2</sub>  
Extruded 10:1 @ 1094°C  
Swaged to 70% R.A. @ 982°C  
by Route 3 Heat Treat  
10.5 hours @ 1205°C.

Mag. 10,000 X

FIGURE 44

STRESS TO RUPTURE STRENGTH OF Co-18Cr-20Ni-4.6ThO<sub>2</sub>-PP3-13A-76  
EXTRUDED 10:1 AT 1094°C AND SWAGED 70% R.A. AT 982°C BY ROUTE 3





APPENDIX I

AREAL AND LINEAL ANALYSES PROCEDURES

### AREAL ANALYSIS

(Ref. W. Rostoker and J. Dvorak, "Interpretation of Metallographic Structures," 1965, pp. 195-219, Academic Press, N.Y.)

#### PROCEDURE:

1. Procure photomicrograph of material at a magnification where the average particle size is approximately 0.5 cm.
2. Construct a square or rectangular area so that approximately 100 particles are within that area.
3. Measure the diameter of each of these 100 particles in microns.
4. Total the number of particles (Q<sub>1</sub>, Q<sub>2</sub>....) at each diameter size, that is, at .01  $\mu$ , .02  $\mu$ , .03  $\mu$ , etc. (D<sub>1</sub>, D<sub>2</sub>, D<sub>3</sub>, etc.).
5. Calculate the total area (A<sub>p</sub>) covered by particles

$$A_p = \frac{\pi D_1^2}{4} \times Q_1 + \frac{\pi D_2^2}{4} \times Q_2 + \frac{\pi D_3^2}{4} \times Q_3 + \dots$$

6. Calculate area fraction (F)

$$F = \frac{A_p}{A_t} \quad \text{where } A_t = \text{Area (in } \mu^2 \text{) encompassed by square or rectangle as per Step \#2.}$$

NOTE: F also equals volume fraction [A.B. Wenterbottom in "The Physical Examination of Metals", (B. Chalmers and A. G. Quarrell, editors), Chapter I., Arnold, London, 1960.]

### LINEAL ANALYSIS

(Ref. W. Rostoker and J. Dvorak, "Interpretation of Metallographic Structures", 1965, pp. 195 - 219, Academic Press, N.Y.)

#### PROCEDURE:

1. Procure photomicrograph of material at a magnification where the average dispersoid size is approximately 0.5  $\mu$ m.
2. Impose a transparent grid (series of parallel lines 1 cm. apart) over the area to be measured.
3. Measure (in cm.) the portion of each line which is occupied by a dispersoid. Add all portions together to get  $L_0$ , the total portion of all lines covered by dispersoids.
4. Calculate volume fraction (F)

$$F = \frac{L_0 \text{ (cm)}}{L \text{ (cm)}} \quad \text{where } L_0 = \text{portion of lines covered by dispersoids (determined in Step 3)}$$

$$L = \text{total length of all grid lines imposed on photomicrograph}$$

5. Determine "particles per unit area" ( $N_A$ )
  - a. Count the number of dispersoid particles in the photomicrograph. If less than half a particle lies in the area, do not count it.

- b. Calculate area represented by the photomicrograph in  $\text{cm}^2$  and convert to  $\mu^2$  using the following conversion factor:

$$C. F. = \left( \frac{10,000}{\text{magnif. of Photomicrographs}} \right)^2$$

- c. Calculate  $N_A$ :

$$N_A = \frac{\text{Total particles counted}}{\text{Area in } \mu^2} = \text{particles}/\mu^2$$

6. Calculate "particles per unit length of line" ( $N_L$ )

$$N_L = \sqrt{F \times \frac{3\pi}{8} \times N_A} = \text{particles}/\mu$$

7. Calculate the radius "r" of the dispersoid

$$r = \frac{2}{\pi} \times \frac{N_L}{N_A}$$

8. Calculate the "mean free path" or average distance (D) between the peripheries of particles (dispersoids).

$$D = \frac{1 - F}{N_L}$$

APPENDIX II

NEW TECHNOLOGY

## APPENDIX II

NEW TECHNOLOGY

After a diligent review of the work performed under this contract, NAS3-11162, no new innovation, discovery, improvement or invention was made.

APPENDIX III  
DISTRIBUTION LIST

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